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# Operating Manual

## SPECORD PLUS Accessories
## UV/Vis Spectrophotometer

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# 1 Notes for users

## 1.1 User manual notes

Contents

The operating instructions provide information about the design and operation of the accessories for the following UV/Vis spectral photometers:

- SPECORD 50 PLUS
- SPECORD 200 PLUS
- SPECORD 210 PLUS
- SPECORD 250 PLUS

The operating instructions provide the necessary know-how for the safe handling of the accessories in conjunction with the SPECORD PLUS. The user manual further includes notes on the maintenance and service of the accessories and potential causes and remedies of any faults.

Conventions

**Instructions for actions** which occur in chronological order are numbered and combined in action units.

**Warnings** are indicated by warning triangles and a signal word. The type, source and consequences of the danger are stated together with notes on preventing the danger. The meaning of the signal words used is explained in the chapter "Safety instructions" on p. 9.

The elements of the control and analysis program are indicated as follows:

- Program terms are indicated by small caps (e.g. menu FILE).
- Buttons are shown by square brackets (e.g., [OK])
- Menu items are separated by arrows (e.g. FILE ▸ OPEN).

User requirements

These instructions are aimed at qualified specialist users with knowledge of UV/Vis analysis. The instructions are limited to describing the functionality of accessories for the SPECORD PLUS.

For the safe operation of the accessories, knowledge of the "ASpect UV" and "SPECORD PLUS " operating instructions is also required. Basic knowledge of working with computers are required.

Symbols and signal words used

The user manual uses the following symbols and signal words to indicate hazards or instructions. The warnings are always placed before an action.

### Danger

This signal word indicates high risk hazards. If not avoided, they can lead to death or serious injuries.

**WARNING**

This signal word indicates medium risk hazards. If not avoided, they can lead to death or serious injuries.

**CAUTION**

This signal word indicates low risk hazards. If not avoided, they can lead to minor or moderate injuries.

**ATTENTION**

Provides information on potential material or environmental damage.

## 1.2 Safety instructions

The general safety instructions for the SPECORD PLUS basic equipment apply (→ Manual "SPECORD PLUS", section "Safety instructions").

Note the following:

- Always use the packaging supplied for storage and transport!
- Instructions applying specifically for an accessory item are listed in the corresponding chapter.
- Never touch the optical mirrors of the accessories with your fingers!
- Avoid pollution of the sample chamber and accessories with strongly corrosive substances! If working with strongly corrosive or volatile substances use vapor-proof cells with sealing plugs.
- Observe the operating instructions and safety notices for the third party system components supplied (e.g. liquid thermostats).

## 1.3 Maintaining accessories

The accessories are predominantly maintenance-free. The following applies to all accessories:

- Avoid contamination by handling sample substances with care.
- Wipe spilled samples or reagents immediately with an absorbent cloth.
- Do not expose accessories to a corrosive atmosphere as they might corrode.
- Where applicable, observe the additional care instructions for the individual accessory items.

## 1.4 The sample chamber design of the SPECORD PLUS

The measuring and reference beam enter the sample chamber of the SPECORD 200/210/250 PLUS from the rear. In the SPECORD 50 PLUS, only the measuring beam enters the sample chamber in the middle from the rear. The reference beam is projected onto a receiver in the photometer chamber. One or two windows, respectively, protect the photometer chamber against dust and contamination from reagents.

Cell holders for standard cells are slid directly into the adapter plates on the front sample chamber wall and are near to the receiver. Cells with cloudy samples of 10 mm pathlength are positioned in special cell ducts directly in front of the receiver.

Cell changer, sipper and inserts for measuring the transmission, reflection and remission of solid, paste-like and liquid samples are fitted onto the support rods. The removable side components of the sample chamber can be replaced easily and adapted for various accessories. In the sides of the sample chamber there are apertures of various sizes through which the sample hose of the cassette sipper system, electrical supply cables for accessories and optical fiber cables of external sample sensors are fed. In the front sample chamber wall there are two apertures for tempering and waste hoses. In the right sample chamber wall are the electrical connection sockets for the accessories and the identification connectors of the accessories.

In the SPECORD 200/210/250 PLUS, the left beam path (**M**) is by default intended for sample measurement and the right beam path (**R**) is intended for reference measurement. The description of the installation of the accessories refers to this default configuration.

If the beam paths were swapped in the software, this must be taken into account during the installation of the accessories.

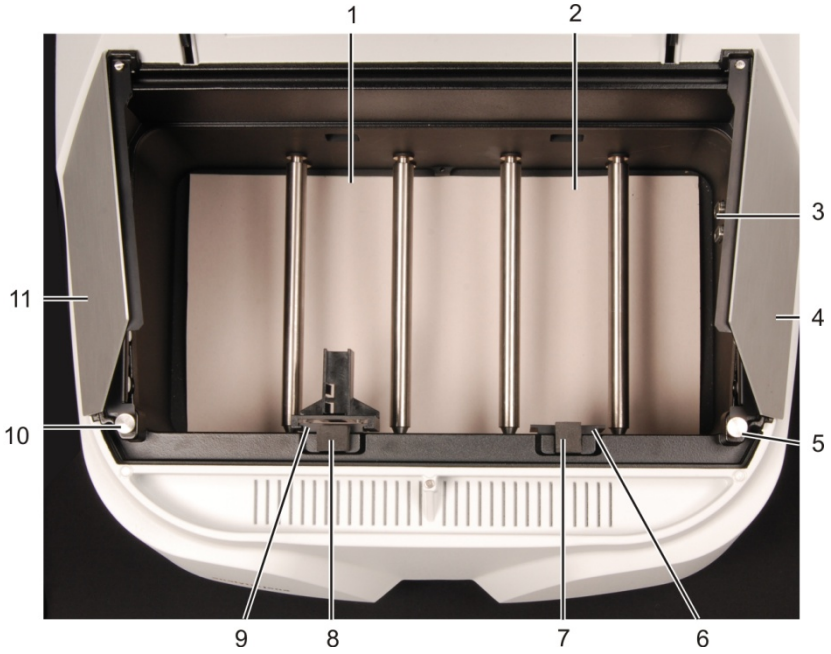



**Fig. 1 Sample chamber of the SPECORD 200/210/250 PLUS**

| | | | |
|---|---|---|---|
| 1 | Measuring channel | 5, 10 | Attachment screws for side components |
| 2 | Reference channel | 6, 9 | Plates to accept the cell holders |
| 3 | Connection sockets for accessory connectors | 7, 8 | Cell ducts to accept turbid samples |
| 4, 11 | Removable side components | | |

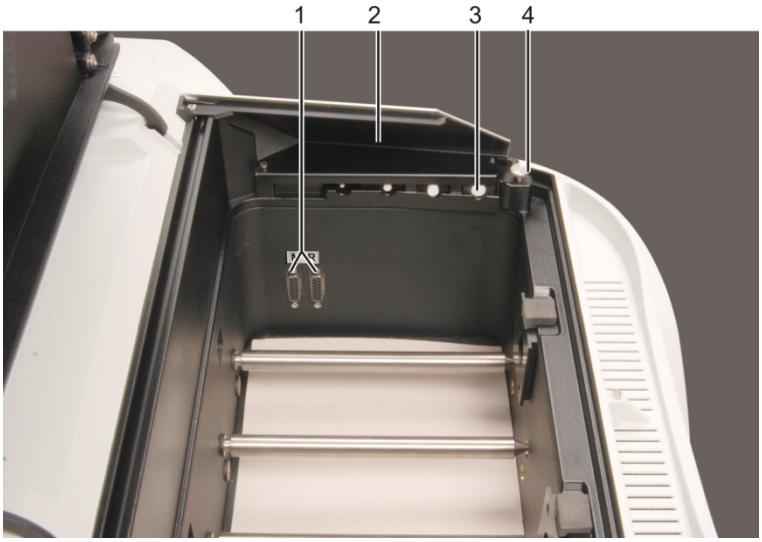



**Fig. 2 Right sample chamber wall with connection sockets**

| | | | |
|---|---|---|---|
| 1 | Connection sockets for accessory connectors | 3 | Apertures for accessory hoses and cables |
| 2 | Removable side component | 4 | Side component attachment screw |

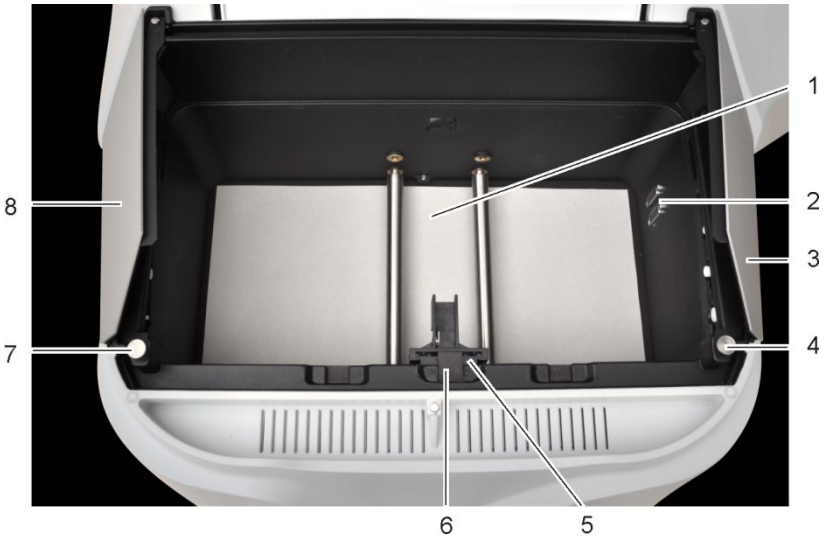



**Fig. 3 Sample chamber of the SPECORD PLUS**

1 Measuring channel
2 Connection sockets for accessory connectors
3,8 Removable side components
4,7 Side component attachment screws
5 Plate to accept the cell holder
6 Cell duct to accept turbid samples

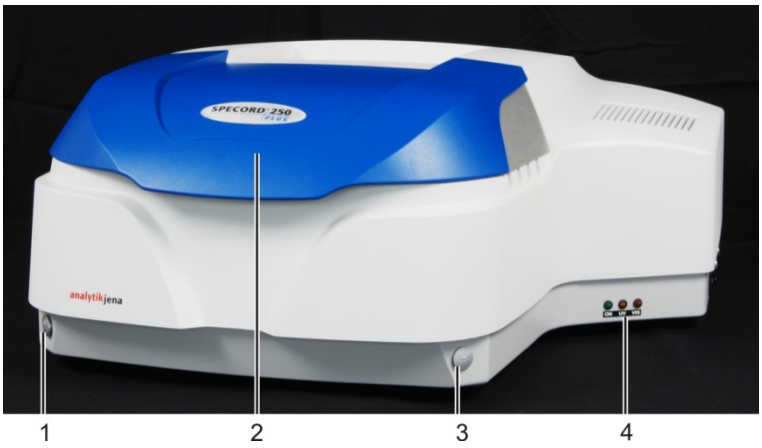



**Fig. 4 Front connections at the SPECORD PLUS**

1, 3 Apertures in the front sample chamber wall
2 Sample chamber cover
4 Status lamps for mains voltage, halogen and deuterium lamps

## 1.5 Converting the sample chamber

Converting the support rods

The accessories require two different installation heights in the sample chamber. The support rods must be converted accordingly.

1. Unscrew the support rods (rotate counterclockwise).
2. Slide one support rod forward and remove it from the sample chamber.
3. Insert the support rod into the bottom or top aperture in the front sample chamber wall. Place the support rod into the holder at the rear sample chamber wall and screw it back (rotate clockwise).
4. Repeat with the remaining support rods.

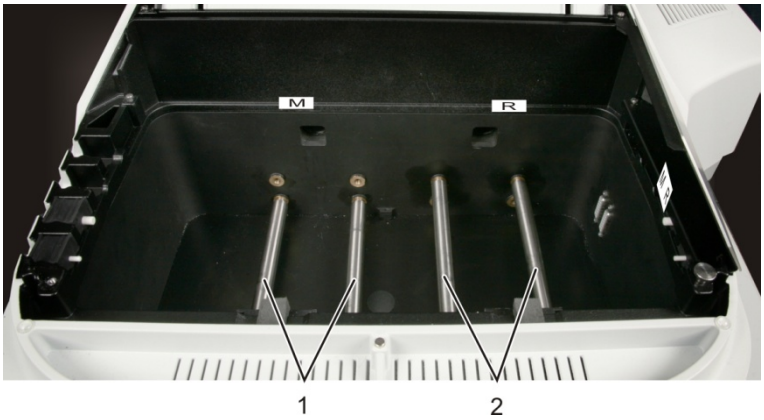



**Fig. 5 Positions of the support rods in the sample chamber**

1 Support rods in the bottom position
2 Support rods in the top position

Attaching the side components

To install various accessories it is necessary to temporarily remove the side components or replace them by different ones.

The side components are attached from three points. On the side of the photometer the V-shaped panel ends are clamped down below two screw heads. On the front side the side component is screwed to the sample chamber wall using the knurled head screw.


1 Knurled head screw
2 V-shaped ends under the screw heads

**Fig. 6 Attaching the side components**

## Attention

Ensure that the side component is positioned correctly!

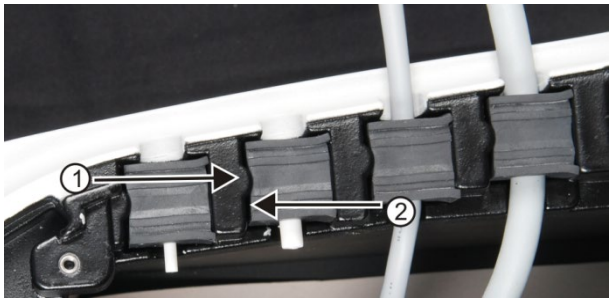
When inserting the side components make sure that the panel ends are pushed under the screw heads and are not bent. If the side component is not positioned correctly, the knurled head screw cannot be screwed on.

Inserting the rubber seals in the apertures of the side walls

The apertures in the side walls are fitted with rubber seals through whose holes electrical cables and sample hoses are routed light-proof. For this reason the shape of the apertures and the rubber seal are matched.

The rubber seals are inserted correctly if

- the slot of the seal points down,
- the smooth edge of the seal (2) points to the curved side of the aperture (1).




1 curved side in the aperture
2 smooth plug side

**Fig. 7 Inserting the rubber seals into the sample chamber apertures**

### Attention

To ensure the light-proof properties, select the rubber seal with the correct cut-out for the cable. The seal must surround the cable as tightly as possible.

## 1.6 Configuring the zeroth order

When the zeroth order is configured, white undispersed light passes through the sample chamber. The zeroth order can be configured separately for the halogen lamp and for the deuterium lamp. The halogen lamp (Vis lamp) is particularly suited for adjusting the accessories, because the intensive beam can easily be observed.

1. Switch on the SPECORD PLUS and start ASpect UV.
2. To configure the zeroth order of the halogen lamp, select INSTRUMENT ▸ ZEROTH ORDER ▸ VIS LAMP.
3. To configure the zeroth order of the deuterium lamp, select INSTRUMENT ▸ ZEROTH ORDER ▸ UV LAMP.

## 1.7 Measuring the reference sample

In the SPECORD 50 PLUS the use of the reference sample is easy.

- Insert the reference sample into the beam path and perform the reference measurement. Replace the reference sample with the sample containing the analyte and perform the sample measurement.

SPECORD 200/210/210 PLUS offer various options for making reference and sample measurements, some of which, unfortunately, can lead to incorrect results.

The following combinations are possible and lead to correct results:

| Combination | Reference measurement | | Sample measurement | |
|---|---|---|---|---|
| | Measuring beam path | Reference beam path | Measuring beam path | Reference beam path |
| 1 | Reference | blank | Sample | blank |
| 2 | Reference | Reference | Sample | Reference |
| 3 | blank | blank | Sample | Reference |

These combination options must also be considered when creating a measurement sequence for the cell changers (→"Measurement with the cell changer" p. 49).

## 1.8 Using accessories in ASpect UV

Electrically controlled accessories and accessories that are connected to the SPECORD PLUS by means of an identification connector, is automatically detected and displayed in the ASpect UV method parameters when the spectrometer is switched on. The accessory-specific method settings are described in the following chapters for the respective accessories.

Passive accessories are accessories that are not electronically controlled or automatically detected via an identification plug:

- Standard cell holder
- Cell holder, 100 mm
- Holder for round cells
- Cell holder, 10 mm, thermostated
- Cell holder, 50 mm, thermostated
- Holder for microcells
- Adjustable cell holder for microcells
- Adjustable holder, 10 to 50 mm
- Holder for cylindrical cells
- Universal holder for accessories
- Fiber coupling with measuring sensor

The passive accessories can be selected in all modules of ASpect UV under the method parameters, on the ACCESSORY tab.

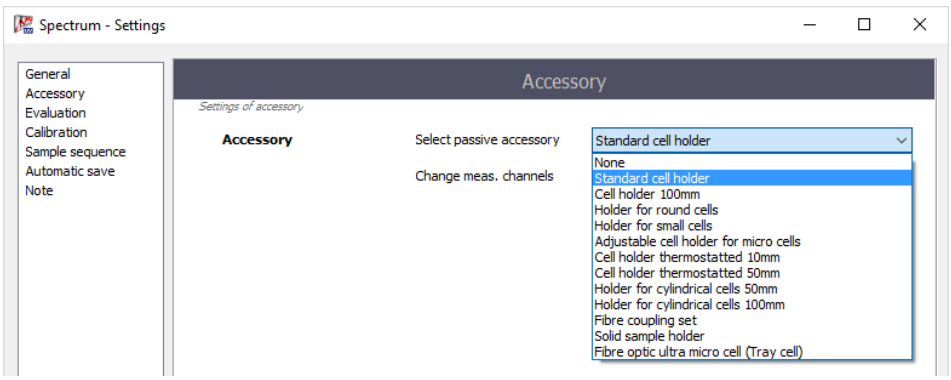



**Fig. 8 Selection of passive accessories in ASpect UV**

The accessory is then displayed under the method parameters. However, the selection does not affect the method parameters any further.

## 1.9 Swapping beam paths in the sample chamber.

The beam paths in the sample chamber can be swapped for working with all accessories. The beam path for the sample measurement (M) becomes the reference beam path (R) and vice versa.

For the following accessories, the CHANGE MEAS. CHANNELS option is available to all modules on the ACCESSORY measurement parameter tab:

- All passive accessories without identification connector
- Cassette sipper system
- Autosampler (APG)

## 1.10 Switching off accessories prior to removing

**Attention**

To avoid the danger of short circuit, install or remove electrically controlled accessories only while the SPECORD PLUS is switched off, or switch them off in ASpect UV before removing them from the sample chamber.

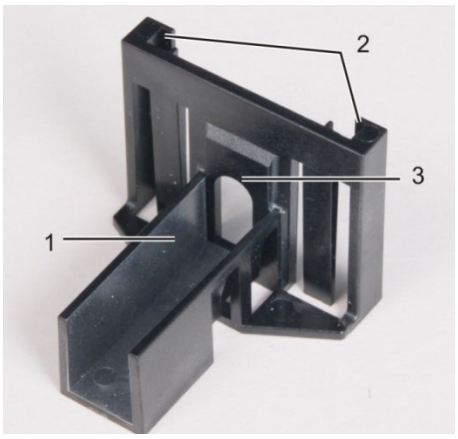
**Note:** This function is not available in the software unless the accessory has already been started, e.g. during a measurement.

- In the ASpect UV main window, select INSTRUMENT ▸ ACCESSORY ▸ ACCESSORY OFF.
  - ✓ The accessory is switched off and will no longer be displayed in the method parameters. You can remove it.
- In the ASpect UV main window, select INSTRUMENT ▸ ACCESSORY ▸ ACCESSORY ON to re-activate the accessory..

# 2 Standard cell holder, 50 mm

The standard cell holder is suitable for cells with a pathlength of up to 50 mm and a width of 12.5 mm. The cells are radiated at a height of approx. 5 to 15 mm above the support level of the cell holder.

Layout

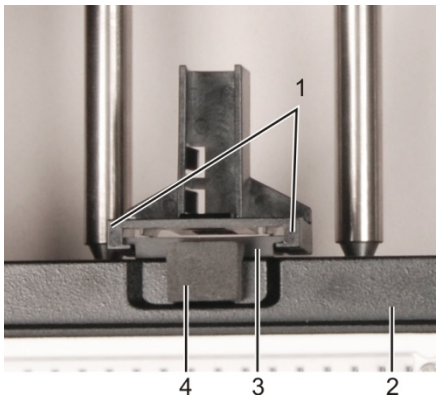



1 Cell adapter
2 Guide
3 Contact surface for cells

**Fig. 9 Standard cell holder, 50 mm**

Installing the cell holder in the SPECORD PLUS

1. Slide the cell holder with the guide onto the adapter plate in the front sample chamber wall in the measuring channel.
2. Slide the second cell holder onto the adapter plate in the reference channel.




1 Guide at the cell holder
2 Front sample chamber wall
3 Adapter plate
4 Sealed measuring position for turbid samples

**Fig. 10 Installing the cell holder in the SPECORD PLUS**

Inserting the cell correctly

1. Fill the cell with the sample at least 20 mm high.
2. Insert the cell tight against the contact surface of the cell holder (arrow in figure below). The optical surfaces of the cell (bare surfaces) are vertical to the measuring or reference beam path and are radiated.

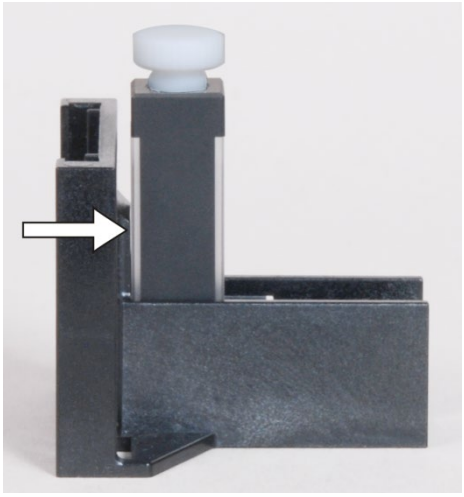


**Fig. 11 Cell inserted correctly in the cell holder**

## Attention

One of the decisive factors for extinction measurement is the pathlength. It is therefore important to place all samples (sample and reference) during a measurement into the same position and orientation. This helps to avoid measuring errors caused by wedge or angle errors or the parallel offset of the radiation.

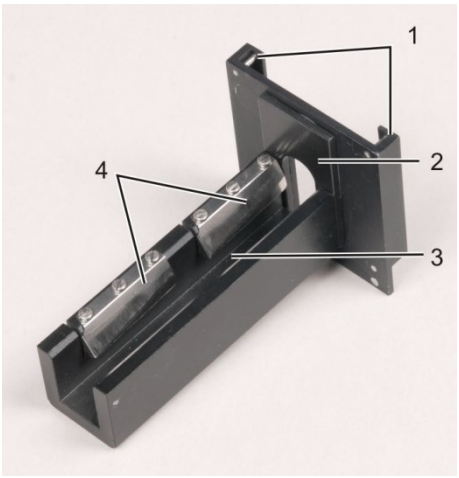
# 3 Cell holder, 100 mm

The cell holder is designed for standard cells with the following dimensions:

| | |
|---|---|
| Pathlength | 1 – 100 mm |
| Cell width | 12.5 mm |

The cells are radiated at a height of approx. 5 to 15 mm above the support level of the cell holder.

Layout




1 Guide
2 Contact surface for cells
3 Cell adapter
4 Leaf springs

**Fig. 12 Cell holder, 100 mm**

Installing the cell holder in the SPECORD PLUS

1. Slide the cell holder with the guide onto the adapter plate in the front sample chamber wall in the measuring channel.
2. Slide the second cell holder onto the adapter plate in the reference channel.

Inserting the cell correctly

Observe the notes in section "Standard cell holder, 50 mm" p. 14.

# 4 Cell holder, 10 mm, thermostated

In this cell holder, the temperature is controlled by means of a liquid thermostat.

Optionally the thermostated cell holder can be equipped with a magnetic stirrer so that an even temperature distribution in the cell is achieved more quickly. Optimum stirring of the sample is achieved when using magnetic stirring rods with a diameter of 2 mm and a length of 5 mm. For cells with cylindrical stirring bottom, it is also possible to use magnetic stirring rods with a diameter of 3 mm and a length of 5 mm. However, the stirring power is not sufficient to homogeneously distribute e.g. reagents in the sample.

The thermostated cell holder can be combined with other sample holders:

- As a holder for reference samples whilst in the measuring channel the thermostated 6- or 8-cell changers are being used. Both cell holders or changers can be temperature-controlled using the same thermostat.
- As thermostated holder for flow cells in conjunction with the cassette sipper system

The cell holder is designed for standard cells with the following dimensions:

| | |
|---|---|
| Pathlength | 10 mm |
| Cell width | 12.5 mm |
| Minimum filling height | 20 mm |
| Radiation height | 5 – 15 mm |
| Temperature range | up to 100 °C |
| Inside hose diameter | 4 mm |
| Outside hose diameter | 6 mm |

Commercially available cells made of glass, quartz or plastic as well as macro and semi-microcells with cylindrical stirring bottom can be used. Owing to the power limits of the magnetic stirrer, only cells with a flat bottom and a bottom thickness of no more than 1.5 mm are suited if the stirring function is used.

The stirring speed is set at the temperature control unit. The stirring power must be increased gradually in order to avoid jamming of the magnetic stirrer.

Layout

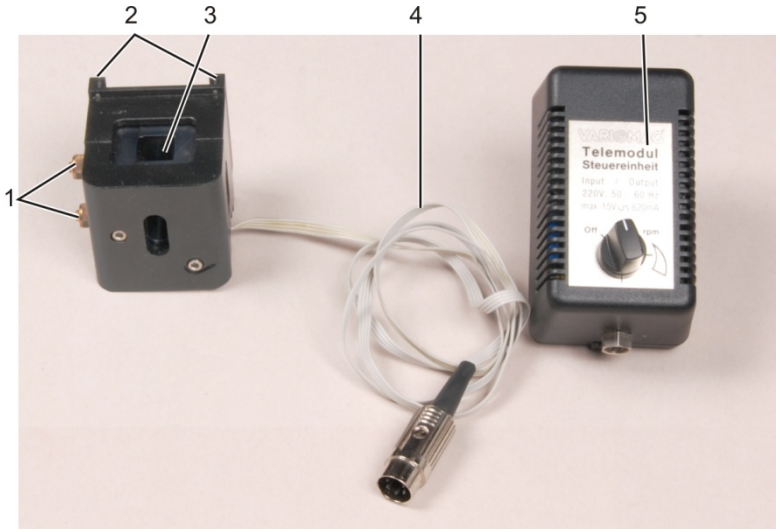



**Fig. 13 Thermostated cell holder with 10 mm pathlength**

1 Tube couplings for temperature control hoses
2 Guide
3 Cell duct
4 Cable with connector for magnetic stirrer
5 Control unit for magnetic stirrer

Inserting the magnetic stirrer

The magnetic stirrer is an optional accessory. It can be retrofitted:

1. Undo the two knurled head screws at the bottom of the cell holder and remove the floor plate.
2. Insert the coil of the magnetic stirrer into the square aperture at the floor of the cell holder. The ribbon cable must be aligned with the base plate.
3. Attach the floor plate using the two knurled head screws. The spacer block on the floor plate, which implements the correct cell height without the stirrer inserted, must point outward.

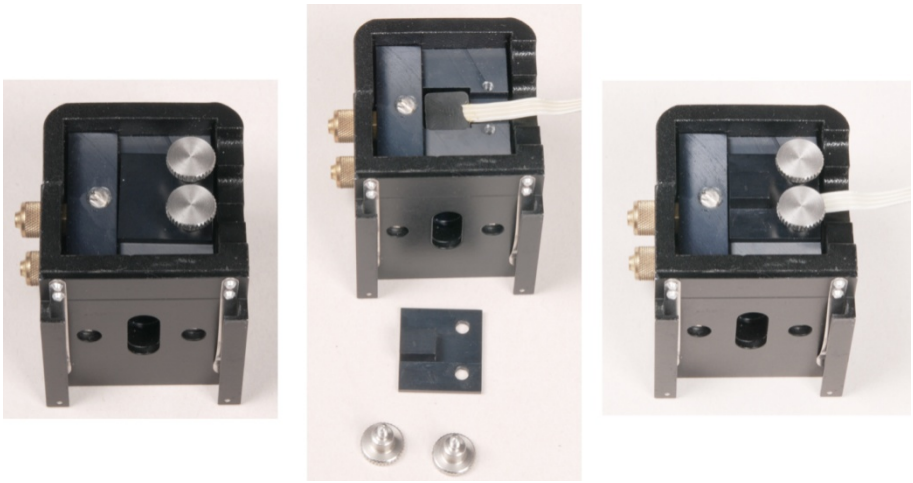


**Fig. 14 Retrofitting the magnetic stirrer**

Installing the thermostated cell holder in the sample chamber

**Note:** Lay the supply cables for the connections to SPECORD PLUS, the water thermostats and the magnetic stirrer, making sure they do not protrude into the beam path in the sample chamber.

1. Remove the two cover caps from the apertures of the bottom front panel of the device (1 and 3 in Fig. 4 p. 9).
2. Slide the cell holder onto the adapter plate in the measuring channel.
3. Feed the hoses below the support rods through the apertures to the outside. To increase light-proofing the apertures in the sample chamber wall have been provided with steps. Should the hose get stuck at these steps when feeding it through, free it up by rotating it slightly. If it is already visible in the outer aperture, bend it towards the aperture with e.g. a pen.
4. When using the magnetic stirrer:
   - Detach one side component of the sample chamber.
   - Remove one rubber seal from one aperture in the sample chamber wall.
   - Magnetic stirrer with round cable: Remove the white plug from the seal and insert the cable into the seal.
   - Magnetic stirrer with ribbon cable: Place the cable flat into the aperture.
   - Insert the rubber seal into the aperture with the slot pointing down.
   - Re-attach the side component.
   - Plug the connector of the magnetic stirrer into the socket of the control unit.
   - Connect the control unit to the mains supply.

Inserting the cell

1. Fill the cell with the sample at least 20 mm high.
2. When using the magnetic stirrer: Insert a magnetic rod into the cell.
3. Insert the cell into the aperture of the cell holder. The optical surfaces of the cell (bare surfaces) are vertical to the measuring or reference channel and are radiated.
4. Set the stirring frequency at the control unit using the rotary knob. Gradually increase the stirring frequency to avoid jamming of the magnetic stirrer.

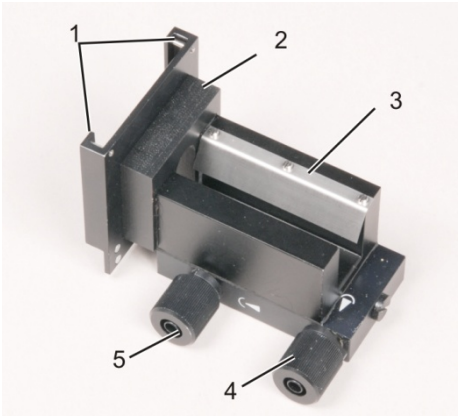
# 5 Cell holder, 50 mm, thermostated

In this cell holder, the temperature is controlled by means of a liquid thermostat.

The cell holders are designed for standard cells with the following dimensions:

| | |
|---|---|
| Pathlength | up to 50 mm |
| Cell width | 12.5 mm |
| Minimum filling height | 20 mm |
| Radiation height | 5 – 15 mm |
| Temperature range: | up to 100 °C |
| Inside hose diameter: | 4 mm |
| Outside hose diameter: | 6 mm |

Layout




1 Guide
2 Contact surface for cells
3 Cell adapter with leaf spring
4, 5 Tube couplings for temperature control

**Fig-15 Thermostated cell holder, 50 mm**

Installing the thermostated cell holder in the sample chamber

Note: Lay the supply cables for the connections of the water thermostat to the SPECORD PLUS, making sure they do not protrude into the beam path in the sample chamber.

1. Remove the two cover caps from the apertures of the bottom front panel of the device (1 and 3 in Fig. 4 p. 9).
2. Slide the cell holder onto the adapter plate in the measuring channel.
3. Feed the hoses below the support rods through the apertures to the outside.

   To increase light-proofing the apertures in the sample chamber wall have been provided with steps. Should the hose get stuck at these steps when feeding it through, free it up by rotating it slightly. If it is already visible in the outer aperture, bend it towards the aperture with e.g. a pen.
4. Connect the hoses to the thermostat.

Inserting the cell

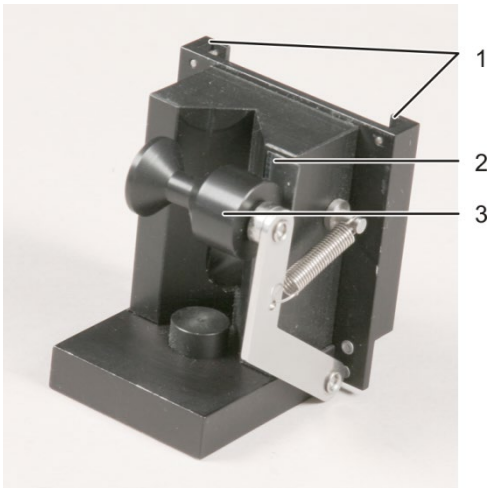
Observe the notes in section "Standard cell holder, 50 mm" p. 14.

# 6 Holder for round cells

The holder is used to accept round cells or ampules (cell tests) or reagent tubes.

| | |
|---|---|
| Cell diameter | 11 mm to 16 mm |
| Cell height | 40 mm to 70 mm |
| Minimum filling height | 20 mm |

Layout




1 Guide
2 Prismatic contact surface
3 Spring rocker

**Fig. 16 Round cell holder**

The round cells are pressed by the spring rocker into the prismatic cell adapter.

Installing the round cell holder in the sample chamber

1. Slide the round cell holder with the guide onto the adapter plate in the front sample chamber wall in the measuring channel.
2. If required, position a second cell holder onto the adapter plate in the reference channel.

Inserting the round cell

1. Pull the spring rocker back.
2. Place the round cell into the adapter.
3. Carefully put the spring rocker back in place.
   ✓ The round cell is positioned correctly by the prismatic contact surfaces.

## Attention

Some manufacturers of cell tests place a line marking on the round cells. These round cells must be positioned in the cell holder in such a way that the marking is exactly in the measuring beam direction.

If this marking is missing, you should carry out multiple measurements and rotate the cell in between the measurements to avoid pathlength errors during high precision measurements.

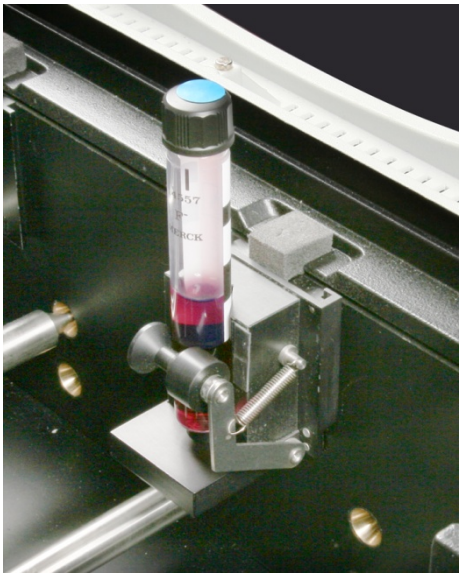


**Fig. 17 Round cell holder installed in the SPECORD PLUS**

# 7 Holder for microcells (variable pathlength)

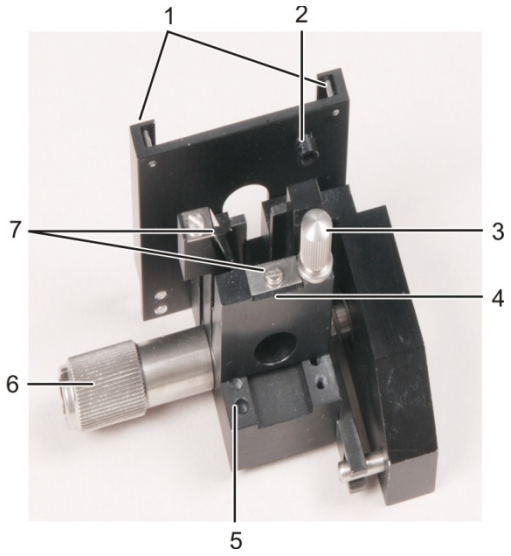
The holder for microcells is adjustable and can accept cells with different pathlength.

The holder is designed for cells with the following dimensions:

| | |
|---|---|
| Pathlength | 1, 2, 5 and 10 mm |
| Cell width | 12.5 mm |
| Radiation height | 8.5 mm |

If cells with non-blackened rims are used, the cell holder can be installed in the sample chamber in combination with the base plate with aperture baffle (→ "Base plate with aperture baffle" S. 130). The aperture baffle prevents distortion of the measuring result by light that passes through the cell walls or through air bubbles on the cell walls.

Layout




1 Guide
2 Locking screw
3 Knurled head screw for locking the slide
4 Slide
5 Hole positions of the slide
6 Horizontal adjustment screw
7 Leaf springs

**Fig. 18 Holder for microcells with variable pathlength**

Installing the holder for microcells

1. Adjust the slide in accordance with the cell pathlength.
   Unscrew the knurled head screw at the slide (3), place the slide (4) into one of the hole positions (5) provided and fasten it with the knurled head screw.
2. Slightly unscrew the locking screw at the holder (2) to prevent jamming when inserting the holder.
3. Slide the holder onto the adapter for the cells in the sample chamber wall.
4. Tighten the locking screw (2) to secure the holder relative to the beam path.
5. Insert an empty cell or one filled with solvent into the holder. The cell is pressed by the leaf spring (7) into the cell guide.
6. For adjustment, configure the zeroth order of the Vis lamp (→"Configuring the zeroth order" p. 11).
7. Initially, adjust the cell holder visually:

- Monitor through the gap how the light hits the cell. Using the knurled head screw (6 in Fig. 18) adjust the cell in such a way that the light hits the cell at the center.
- In order to see the passing light beam more clearly, insert a paper strip of approx. 10 mm width into the opening for cloudy samples and observe the traced light beam from above.

8. In the PHOTOMETRY module set a method with the following parameters:

   GENERAL tab:

| Parameter | |
|---|---|
| MEAS. MODE | TRANSMITTANCE |
| WAVELENGTHS [NM] | 500.00 |
| INTEGRATION TIME [S] | 0.1 |
| SLIT [nm]<br>(SPECORD 210/250 PLUS only) | 1 |

   SAMPLE SEQUENCE tab:

   - One reference measurement in first position
   - Further sample rows with the sample type SAMPLE for adjustment

9. Start the measurement with  .
10. Perform the reference measurement.
11. Adjust the cell holder transverse to the beam path. During repeated measurements, move the cell adapter with the aid of the knurled head screw (6), measuring the transmission **with the sample chamber cover closed**. Repeat the process until a maximum transmission value is reached.

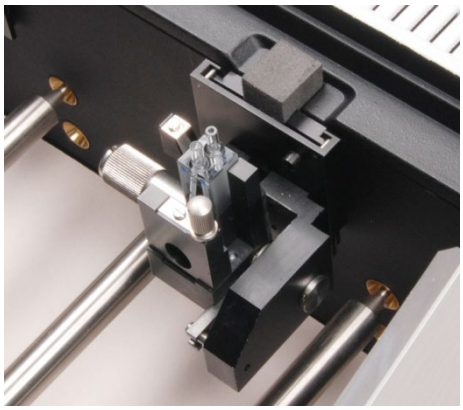


**Fig. 19 Holder for microcells (variable pathlength) installed in the SPECORD PLUS**

Inserting the cell

1. Fill the cell with the sample.
2. Insert the cell into the cell holder. The optical surfaces of the cell (bare surfaces) are vertical to the measuring or reference channel and are radiated.

   ✓ The cell is held reproducible by the lateral spring and the spring on the slide.

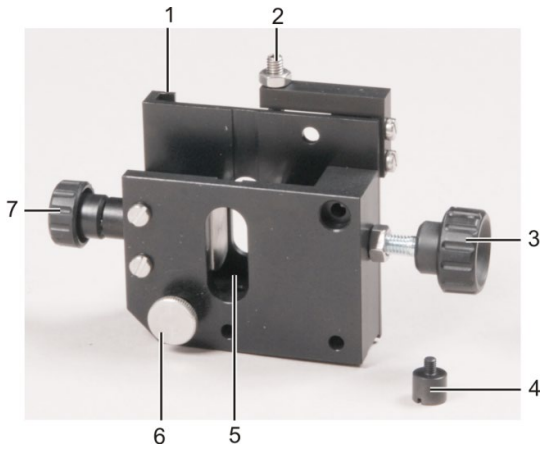
# 8 Adjustable cell holder for microcells

The holder for microcells is adjustable and can accept cells with radiation heights of 8.5 and 15 mm. The holder is designed for cells with the following dimensions:

| | |
|---|---|
| Pathlength | 10 mm |
| Cell width | 12.5 mm |
| Radiation height | 8.5 or 15 mm |

If cells with non-blackened rims are used, the cell holder can be installed in the sample chamber in combination with the base plate with aperture baffle (→ "Base plate with aperture baffle" S. 130). The aperture baffle prevents distortion of the measuring result by light that passes through the cell walls or through air bubbles on the cell walls.

Layout




1 Guide
2 Vertical adjustment screw
3 Horizontal adjustment knurled head screw
4 Headed dowel to adjust the radiation height
5 Cell duct
6 Knurled head screw for fastening the holder
7 Spring catch

**Fig. 20 Adjustable holder for microcells with variable radiation height**

Installing the holder for microcells

1. Depending on the radiation height of the microcell used, fit the headed dower in the cell duct:

| | |
|---|---|
| Radiation height 15 mm: | Remove the headed dowel |
| Radiation height 8.5 mm: | Screw the headed dowel in finger-tight |

2. Slightly unscrew the locking screw at the holder (6) to prevent jamming when inserting the holder.
3. Slide the holder onto the adapter for the cells in the sample chamber wall.

Adjusting the cell holder

4. For adjustment, configure the zero order of the Vis lamp (→"Configuring the zeroth order" p. 11).
5. Insert an empty cell or one filled with solvent into the holder. Pull out the handle a little and place the cell into the duct. The spring in the handle presses the cell against the opposite stop.
6. Initially, adjust the cell holder visually:
   - Monitor through the gap how the light hits the cell. Using the knurled head screw, adjust the cell in such a way that the light hits the cell at the center.

- In order to see the passing light beam more clearly, you can insert a paper strip of approx. 10 mm width into the opening for cloudy samples and observe the traced light beam from above.

7. In the PHOTOMETRY module set a method with the following parameters:

   GENERAL tab:

| Parameter | |
|---|---|
| MEAS. MODE | TRANSMITTANCE |
| WAVELENGTHS [NM] | 500.00 |
| INTEGRATION TIME [S] | 0.1 |
| SLIT [nm]<br>(SPECORD 210/250 PLUS only) | 1 |

   SAMPLE SEQUENCE tab:

   - One reference measurement in first position
   - Further sample rows with the sample type SAMPLE for adjustment

8. Start the measurement with  .
9. Perform the reference measurement against air.
10. Insert the cell into the cell holder. Consecutively modify the vertical adjustment (by rotating the adjustment screw 2 in Fig. 20) and the horizontal adjustment (by rotating the knurled head screw 3 in Fig. 20) and each time measure the transmission **with the sample chamber cover closed**. Repeat the process until a maximum transmission value is reached.

    **Note:** Do not modify the vertical adjustment and the horizontal adjustment of the cell holder at the same time.

11. Tighten the knurled head screw (6 in Fig. 20) to secure the holder relative to the beam path.

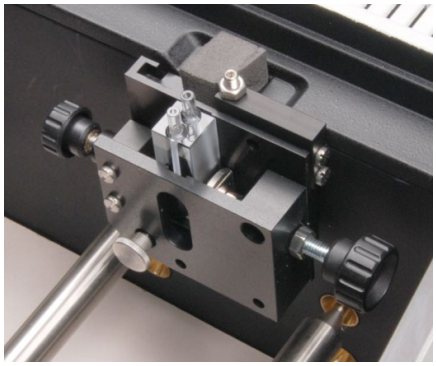


**Fig. 21 Holder for micro cells (adjustable) installed in the SPECORD PLUS**

Inserting the cell

1. Fill the cell with the sample.
2. Pull out the handle a little and place the cell into the cell holder. The optical surfaces of the cell (bare surfaces) are vertical to the measuring or reference channel and are radiated.

   ✓ The cell is pressed reproducible against the contact surface by the spring.

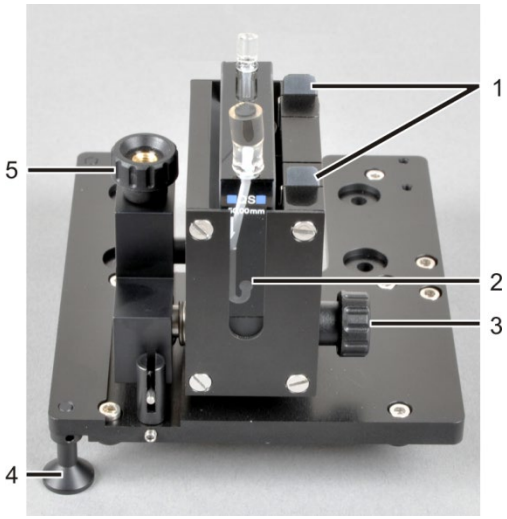
# 9 Adjustable holder, 10 to 50 mm

The holder is adjustable and can accept cells with radiation heights of 8.5 and 15 mm.

The holder is designed for cells with the following dimensions:

| | |
|---|---|
| Pathlength | 10, 20, 40 and 50 mm |
| Cell width | 12.5 mm |
| Radiation height | 8.5 – 15 mm |

Layout




1 Slider with leaf spring
2 Cell duct
3 Horizontal adjustment screw
4 Foot
5 Vertical adjustment screw

**Fig. 22 Adjustable holder for cells (10 to 50 mm) with variable radiation height**

Installing the holder for cells (10 to 50 mm)

1. Screw the support rods in the sample chamber into the top position (→ "Converting the sample chamber" p. 9).
2. Insert the slider with the leaf spring (10 in Fig. 69) at the location corresponding to the cell pathlength.

   **Note:** For cells with a pathlength of 40 or 50 mm use both sliders in order to align the long cell in parallel with the beam path.
3. Place the cell holder onto the support rods in the measuring channel so that the cell adapter points forward towards the receiver. Press down the cell holder on the right side until it engages on the support rods with a click.
4. Place the cell into the holder.

Using the aperture baffle

The cell holder is fitted with an aperture baffle. The baffle is located on the rear of the cell duct and is attached by means of four screws. It prevents distortion of the measuring result by light that passes through the cell walls or through air bubbles on the cell walls. The baffle is particularly suitable for working with cells that have a large pathlength as well as cells with a small aperture and non-blackened rims. The aperture baffle is adjusted for a radiation height of 15 mm but can be adapted for a radiation height of 8.5 mm.

If cells with large apertures are used, the aperture baffle is removed in order to achieve a higher light level and thus a better signal-to-noise ratio.

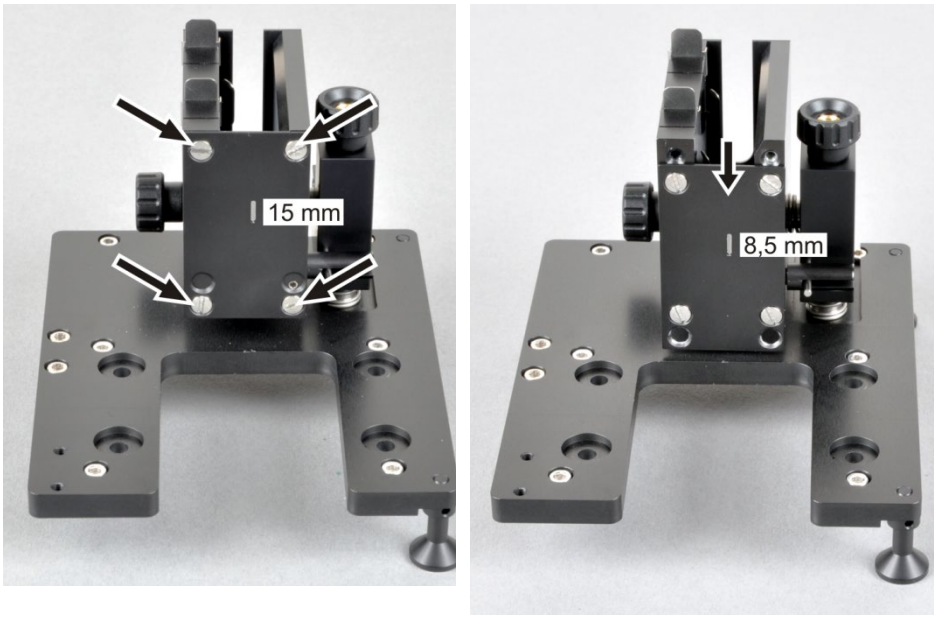



**Fig. 23 Aperture baffle adapted to a radiation height of 15 and 8,5 mm, respectively**

- To remove the aperture baffle: Unscrew the fours screws at the rear of the cell duct and remove the aperture baffle.
- To adapt the aperture baffle to a radiation height of 8.5 mm: Using the four screws, fasten the aperture baffle in the lower position on the rear of the cell duct.

Adjusting the cell holder

1. Configure the zero order of the Vis lamp (→"Configuring the zeroth order" p. 11).
2. If the subsequent measurement is carried out with standard cells or semi-microcells, carry out an adjustment with an empty cell holder.
   If microcells are used for the measurements, place a water-filled microcell in the holder for adjusting.
3. First, visually assess the passage of the undispersed white light through the cell:
   - Insert a paper strip of approx. 10 mm width into the opening for cloudy samples and observe the passing light beam from above.
   - Consecutively turn the screws for vertical and horizontal adjustment (3 and 5 in Fig. 22) until the light hits the paper strip at the center.
4. In the PHOTOMETRY module set a method with the following parameters:

   GENERAL tab:

| Parameter | |
|---|---|
| MEAS. MODE | TRANSMITTANCE |
| WAVELENGTHS [NM] | 500.00 |
| INTEGRATION TIME [S] | 0.1 |
| SLIT [nm]<br>(SPECORD 210/250 PLUS only) | 1 |

SAMPLE SEQUENCE tab:

- One reference measurement in first position
- Further sample rows with the sample type SAMPLE for adjustment

5. Start the measurement with  .
6. Perform the reference measurement.
7. Consecutively change the vertical and horizontal adjustment and each time measure the transmission **with the sample chamber cover closed**. Repeat the process until a maximum transmission value is reached.

   **Note:** Do not modify the vertical adjustment and the horizontal adjustment at the same time.

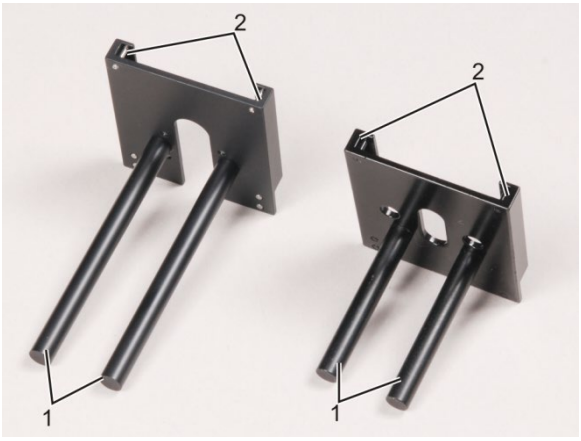
# 10 Holder for cylindrical cells

This holder is used to accept cylindrical cells. It is available in two designs for cells up to 50 mm and 100 mm pathlength.

The holder is designed for cells with the following dimensions:

| | |
|---|---|
| Pathlength | Up to 50 or 100 mm |
| Cell diameter: | 22 mm |

Layout




1 Support rods
2 Guides

**Fig. 24 Holder for cylindrical cells**

Installing the cell holder

Slide the cell holder with the guide onto the adapter plate in the front sample chamber wall in the measuring channel.

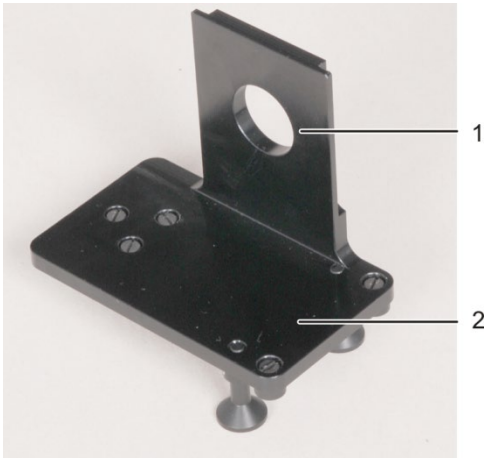
Inserting the cylindrical cell

1. Fill the cell with the sample.
2. Place the cell onto the support rods and slide it against the stop plate of the holder.

**Fig. 25 Holder for cylindrical cells installed in the SPECORD PLUS**

# 11 Universal holder for accessories

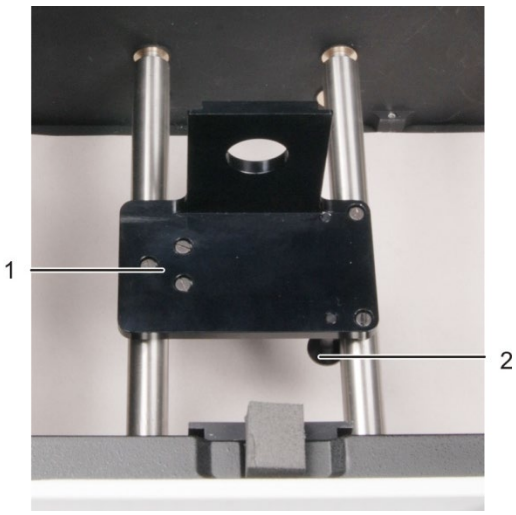
The universal holder is used to accept the holder for the 100 mm absorption tube. Alternatively, it can accept all cell holders that are inserted into the holding plate in the sample chamber wall.




1 Adapter for cell holders
2 Base plate with feet and pressure piece

**Fig. 26 Universal holder**

Installing the universal holder




1 Pressure piece (obstructed)
2 Feet

**Fig. 27 Universal holder installed in the SPECORD PLUS**

1. Place the universal holder onto a pair of support rods. The adapter plate may point forward or back as desired.
2. Press the base plate down.
   ✓ The universal holder engages with a click and is attached onto the support rods.
3. After inserting and fixating the universal holder slide the cell or solid sample holder onto the adapter plate.

# 12 Holder for a 100 mm absorption tube

Absorption tubes are used to measure liquid and gaseous samples. The available holders position the absorption tube optimally in the beam path. In addition, a universal holder is required.

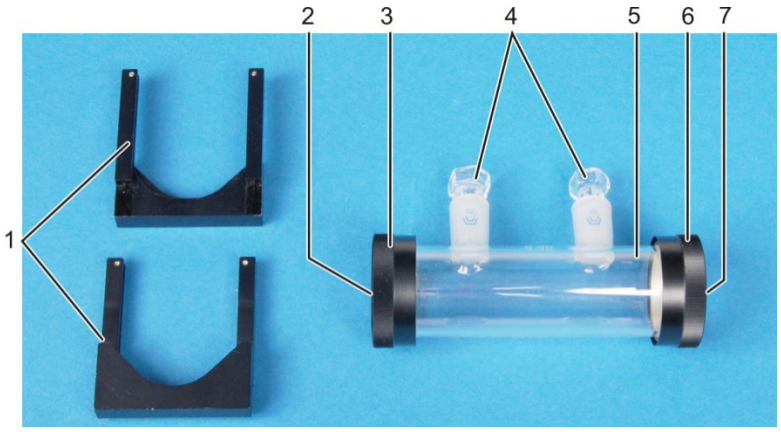



**Fig. 28 Holder and absorption tube**

1 Holder
2, 7 Quartz window
3, 6 Mounts with sealing rings
4 Plugs
5 Glass body of the absorption tube

Inserting the 100 mm absorption tube into SPECORD PLUS

1. Place the universal holder onto a pair of support rods so that the base plate points in the direction of the sample chamber wall.
2. Slide one holder for the absorption tube each from the top onto the adapter plate of the SPECORD PLUS or onto the adapter plate of the universal holder.
3. Move the universal holder to allow the absorption tube to rest on the two holders.
4. Place the absorption tube onto the holders.

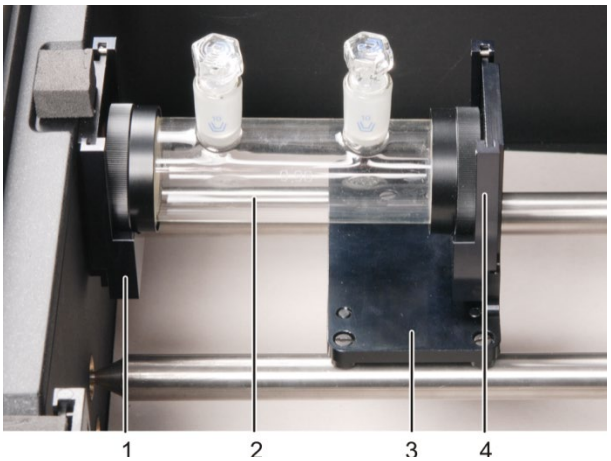



**Fig. 29 100 mm absorption tube**

1, 4 Holders for absorption tube
2 100 mm absorption tube
3 Universal holder

# 13 6-cell changer

The 6-cell changer is an automatic sample changing system for max. 6 standard cells. The linear layout of the cells in the sample block ensures that all cells are radiated with the same optical conditions. This leads to a high measuring accuracy and a very good reproducibility between measuring positions. The 6-cell changer is automatically detected during the device initialization in ASpect UV and displayed in the method parameters.

Design versions

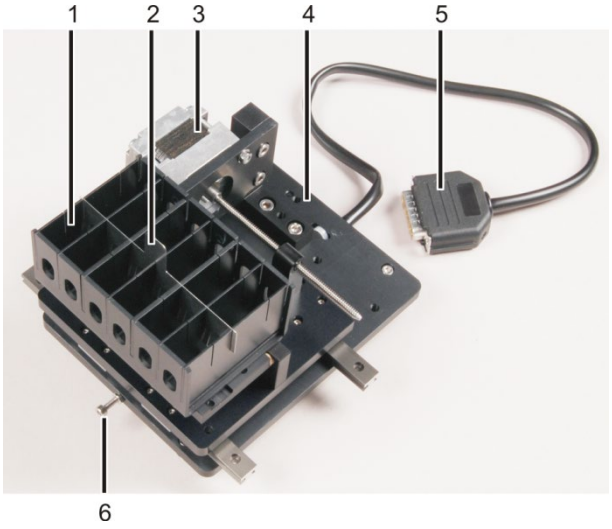
The 6-cell changer is supplied in the following designs:

- Cell changer for cells with variable pathlength, sample block for cells with 10, 20, 40 and 50 mm pathlength, non-thermostated, without stirrer
- Cell changer for cells with 10 mm pathlength, thermostated via external thermostats
- Cell changer for cells with 10 mm pathlength, thermostated via external thermostats and magnetic stirrer
- Peltier-tempered cell changer with and without stirrer
  (→ "Peltier tempered 6- and 8-cell changers" p. 73)

Technical data

| | | |
|---|---|---|
| Cells | Pathlength | 10, 20, 40 and 50 mm |
| | Cell width | 12.5 mm |
| | Minimum filling height | 20 mm |
| | Radiation height | 5 – 15 mm |
| Magnetic stirrer | Power supply | 230 V |

Layout




1 Cell block
2 Slide
3 Step motor
4 Base plate with guide rails
5 Interface cable to SPECORD PLUS
6 Stop bolt

**Fig. 30 6-cell changer for cells with variable pathlength**

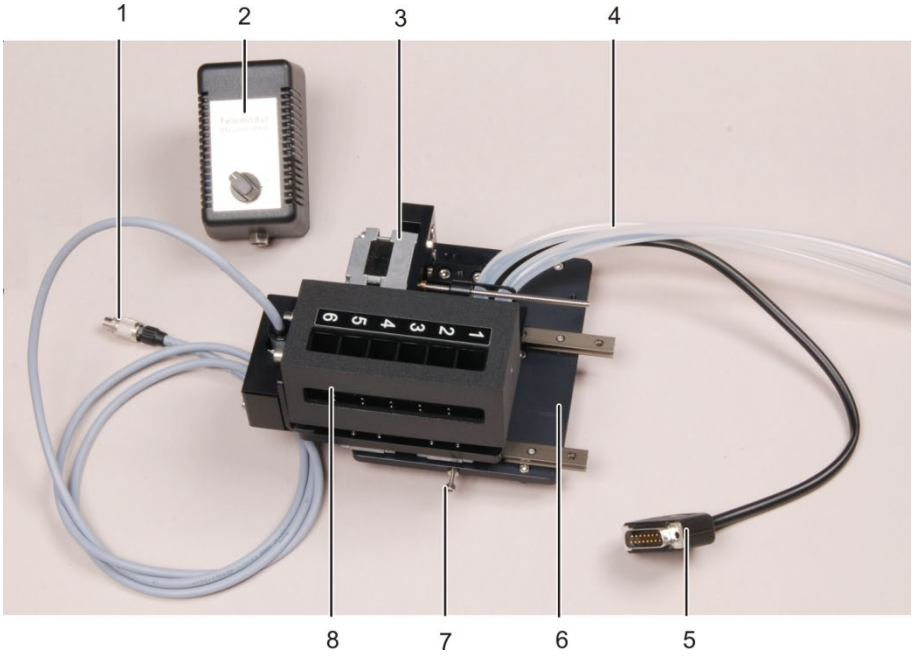



**Fig. 31 6-cell changer, thermostated, with magnetic stirrer**

1 Connection for magnetic stirrer (optional)
2 Control unit for magnetic stirrer (optional)
3 Step motor
4 Hoses for thermostat connection (optional)
5 Interface cable to connect to the SPECORD PLUS
6 Base plate with guide rails
7 Stop bolt
8 Cell block with insulation

Notes on the magnetic stirrer

In combination with the magnetic stirrer, commercially available 10 mm macro cells made of glass, quartz or plastic as well as macro and semi-microcells with cylindrical stirring bottom can be used. Owing to the power limits of the stirrer, only cells with a flat bottom and a bottom thickness of no more than 1.5 mm are suited.

The magnetic stirrer ensures that an even temperature distribution within the cell is reached more quickly. However, the stirring power is not sufficient to homogeneously distribute e.g. reagents in the sample. The stirring power must be increased gradually in order to avoid jamming of the magnetic stirrer.

The stirring speed is set at the separate control unit. Optimum stirring of the sample is achieved when using magnetic stirring rods with a diameter of 2 mm and a length of 5 mm. For cells with cylindrical stirring bottom, it is also possible to use magnetic stirring rods with a diameter of 3 mm and a length of 6 mm.

## 13.1 Installing, removing and adjusting the 6-cell changer

### 13.1.1 Installing the 6-cell changer

This section describes the installation of a thermostated 6-cell changer with magnetic stirrer. If your cell changer does not feature the temperature control or stirrer, skip the respective items in the description.

## Attention

Lay the supply cables for the connections to the SPECORD PLUS, the water thermostats and the magnetic stirrer, making sure they do not protrude into the beam path in the sample chamber.

Lay the water hoses to the thermostats without tension! The cell block of the cell changer must move freely.

The following steps must be carried out:

- Positioning of support rods
- Connecting the thermostat
- Connecting the cell changer
- Connecting the magnetic stirrer
- Optionally, install the aperture baffle


1 Removable side component
2 Connection to the control unit for magnetic stirrer
3 Water hoses
4 Base plate of the cell changer
5 Interface cable to connect to SPECORD PLUS

**Fig. 32 6-cell changer in the SPECORD PLUS**

Positioning of support rods

Screw the support rods in the sample chamber into the lower position (→ "Converting the sample chamber" p. 9).

Connecting the thermostat

1. Remove the cover caps from the apertures of the sample chamber in the front panel of the device (1 & 3 in Fig. 4 p. 9).
2. Feed the hoses below the support rods through the apertures to the outside.

To increase light-proofing the apertures in the sample chamber wall have been provided with steps. Should the hose get stuck at these steps when feeding it through, free it up by rotating it slightly. If it is already visible in the outer aperture, bend it towards the aperture with e.g. a pen.

3. Connect the hoses to the thermostat.

Inserting the cell changer

1. Place the cell changer onto the support rods in the measuring channel and slide it up to the stop against the front sample chamber wall.
2. Press down the cell changer on the right side until it engages on the support rods with a click.
3. Connect the plug to connector (**M**) in the right sample chamber wall.

Connecting the magnetic stirrer

1. Detach the left side component of the sample chamber (→"Converting the sample chamber" p. 9).
2. Remove one rubber seal from one aperture in the sample chamber wall.
3. Remove the white plug from the seal and insert the cable into the seal.
4. Insert the rubber seal into the aperture with the slot pointing down.
5. Re-attach the side component.
6. Connect the control unit to the mains supply.

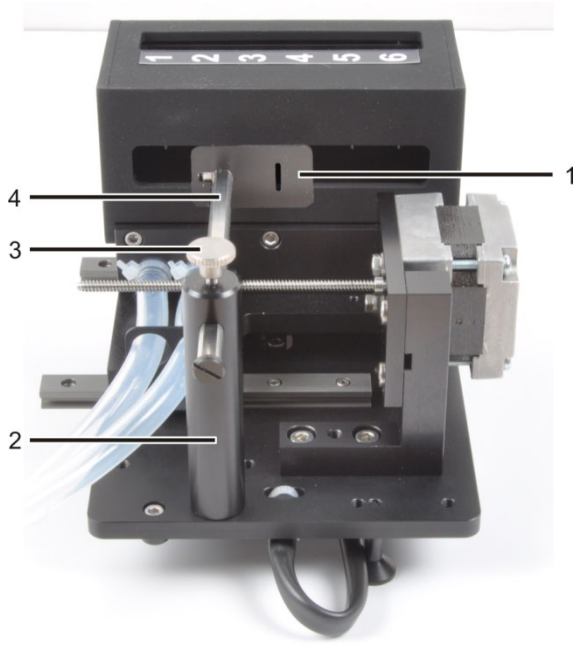
Switch on the SPECORD PLUS.

Switch on the SPECORD PLUS and start ASpect UV.

✓ The SPECORD PLUS is automatically initialized: The monochromator moves, and on the monitor the message "Initialization" is shown. The 6-cell changer is automatically detected and displayed in the method parameters.

### 13.1.2 Using the aperture baffle

If micro cells or semi-microcells made of plastic or glass or quartz cells without blackened rims are used, an aperture baffle needs to be installed on the cell changer (→ "Base plate with aperture baffle" p. 130). The baffle prevents distortion of the measuring result by light that passes through the cell walls or through air bubbles on the cell walls.




1 Aperture baffle
2 Carrier rod
3 Knurled head screw for fastening the baffle mount
4 Baffle mount

**Fig. 33 6-cell changer with aperture baffle**

1. Install the aperture baffle (1) on the rear of the cell changer. To do so, use the hexagon socket wrench (included) to screw the carrier rod (2) to the rear position of the base plate of the cell changer.
2. Loosen the knurled head screw (3) and slide the aperture baffle as close as possible to the cell block. The aperture baffle must neither touch the sample nor contact the cell block when it is moved through the beam path. Tighten the knurled head screw.
3. Adjust the baffle:
   - Configure the zero order of the Vis lamp (→"Configuring the zeroth order" p. 11).
   - Hold a white paper strip as a screen at the later position of the sample.
   - Loosen the knurled head screw (2 in Fig. 108) and slide the baffle until the optical beam hits the sample at the center.

### 13.1.3 Adjusting the 6-cell changer

To position the cells optimally within the beam path the cell changer can be adjusted with the aid of a computer.

Adjusting is required in the following situations:

- first use of the cell changer
- after a basic correction
- after transporting the SPECORD PLUS

**Note:** Before adjusting, wait for 2 hours with lamps switched on.

1. Install the empty cell changer in the sample chamber.

2. Switch the SPECORD PLUS on, start the ASpect UV software and wait until the automatic device initialization is completed.
3. Wait for the warm-up time of 2 hours.
4. Measuring using standard cells or semi-microcells: Perform adjustment with empty cell changer.
   Measurements using microcells for adjusting: Place a water-filled microcell in the each of the 6 positions.
5. Start the automatic adjustment by choosing INSTRUMENT ▸ ACCESSORY ▸ ADJUSTMENT.

### 13.1.4 Removing the 6-cell changer from the sample chamber

Removing the cell changer

1. To move the cell changer to the parking position: Choose INSTRUMENT ▸ ACCESSORY ▸ SAMPLE POSITION / button: [PARKING].
2. To switch off the accessory: Choose INSTRUMENT ▸ ACCESSORY ▸ ACCESSORY OFF.
3. Disconnect the plug from the connector in the right sample chamber wall.
4. Pull up the cell holder on the right side of the base plate until it disengages from the clamp on the support rods with a click.
5. Lift the cell holder out of the sample chamber

Disconnecting the thermostat

1. Disconnect the hoses from the thermostat.
2. Allow the water to drain from the hoses.
3. Pull the hoses from the apertures in the front sample chamber wall.
4. Place the cover caps onto the apertures in the front panel of the SPECORD PLUS (1 & 3 in Fig. 4 p. 9) to restore the light-proofing.

Disconnecting the magnetic stirrer

1. Disconnect the stirrer control unit from the mains. Unscrew the cable and the control unit.
2. Unscrew the side component of the sample chamber.
3. Remove the rubber seal from the aperture in the left sample chamber wall and pull the cable of the magnetic stirrer out of the slot of the rubber seal.
4. Close the opening in the rubber seal with the white plug.
5. Insert the rubber seal into the sample chamber aperture with the slot pointing down.
6. Screw on the side component.

## 13.2 Measurements with the 6-cell changer

The measuring parameter configurations and various operating modes are identical for all cell changers available for the SPECORD PLUS. A detailed description is

available in chapters "Configuring measuring parameters for the cell changer" p. 49 and "Carrying out measurements with the cell changer" p. 50.

## 13.3 Using two 6-cell changers

In the sample chamber of the SPECORD 200/210/250 PLUS two 6-cell changers can be operated simultaneously, one each in the measuring and reference channels.

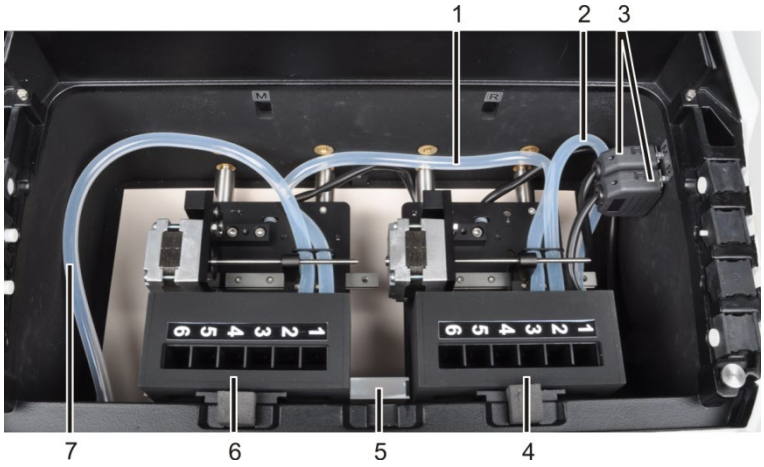



**Fig. 34 Two 6-cell changers in the SPECORD PLUS**

1 Short water hose
2 Water hose (drain)
3 Connector
4 Cell changer in the reference beam path
5 Guide rail
6 Cell changer in the measuring beam path
7 Water hose (feed)

Installing two 6-cell changers in the sample chamber

1. Place the support rods in the bottom position of the reference channel. Install the second cell changer analog to the first changer (→ "Installing the 6-cell changer" p. 34).
2. Using the guide rail (5 in Fig. 34) (included), align both cell changers with respect to each other so that they have the same distance from the front sample chamber. Correct the adjustment of the stop bolts (7 in Fig. 31 p. 34), if required.
3. In case of temperature control by an external thermostat connect the two cell changers with a short piece of hose. Feed the water connection hoses of the cell changers to the outside and connect them to the thermostat.
4. Connect the electrical connection cable for the magnetic stirrer as described above.
   Feed the connection of the cell changer in the reference beam path to the outside through the right sample chamber side. Connect both magnetic stirrers to the control unit using the Y adapter supplied.
5. Adjust the two 6-cell changers in the beam path as described for the 6-cell changer (→ "Adjusting the 6-cell changer" 37).
6. In order to use the adjustment for subsequent measurements, use the adhesive labels (supplied). Attach the "M" and "R" labels to the step motors of the cell changers in the measuring and reference beam paths, making sure they clearly visible.

Measurements with two 6-cell changers

The configuration for the use of two cell changers is found in chapter "Carrying out measurements with two cell changers" p. 53.

# 14 8-cell changer

The 8-cell changer is an automatic sample changing system for eight standard cells.

Design versions

The following additional options are available for the 8-cell changer:

- Magnetic stirrer
- Temperature control using external thermostats
- Peltier tempering with and without magnetic stirrer
  (→ section "Peltier tempered 6- and 8-cell changers" p. 73)
- Hose holder for flow cells or dissolution applications

Cell changers are fully assembled at factory and can only be retrofitted by customer service.

Technical data

| | | |
|---|---|---|
| Cells | Pathlength | 10 mm |
| | Cell width | 12.5 mm |
| | Minimum filling height | 20 mm |
| | Radiation height | 5 – 15 mm |
| Temperature control | By way of external thermostats | |
| Magnetic stirrer | Power supply | 230 V |

Layout

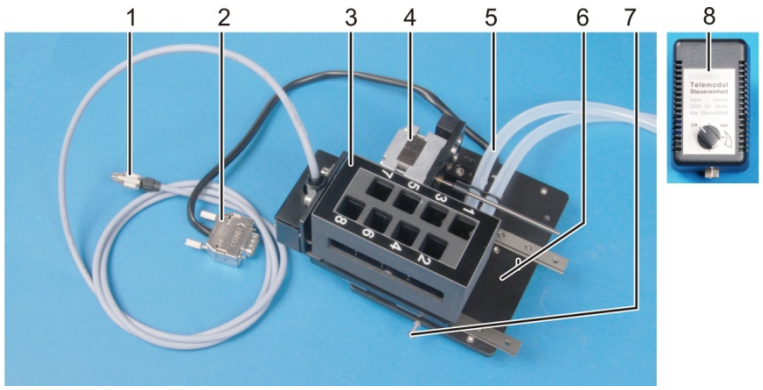



**Fig. 35 Layout of the 8-cell changer**

1 Connection for magnetic stirrer (optional)
2 Interface cable to connect to the SPECORD PLUS
3 Cell block with insulation sleeve
4 Step motor
5 Water hoses for thermostat connection (optional)
6 Base plate with guide rails
7 Stop bolt
8 Control unit for magnetic stirrer (optional)

Notes on the magnetic stirrer

In combination with the magnetic stirrer, commercially available 10 mm macro cells made of glass, quartz or plastic as well as macro and semi-microcells with cylindrical stirring bottom can be used. Owing to the power limits of the stirrer, only cells with a flat bottom and a bottom thickness of no more than 1.5 mm are suited.

The magnetic stirrer ensures that an even temperature distribution within the cell is reached more quickly. However, the stirring power is not sufficient to homogeneously distribute e.g. reagents in the sample. The stirring power must be increased gradually in order to avoid jamming of the magnetic stirrer.

The stirring speed is set at the separate control unit. Optimum stirring of the sample is achieved when using magnetic stirring rods with a diameter of 2 mm and a length of 5 mm. For cells with cylindrical stirring bottom, it is also possible to use magnetic stirring rods with a diameter of 3 mm and a length of 6 mm.

## 14.1 Installing, removing and adjusting the 8-cell changer

### 14.1.1 Installing the 8-cell changer in the sample chamber

This section describes the installation of a thermostated 8-cell changer with magnetic stirrer. If your cell changer does not feature the temperature control or stirrer, skip the respective items in the description.

**Attention**

Lay the supply cables for the connections to the SPECORD PLUS, the water thermostats and the magnetic stirrer, making sure they do not protrude into the beam path in the sample chamber.

Lay the water hoses to the thermostats without tension! The cell block of the cell changer must move freely.

The following steps must be carried out:

- Positioning of support rods
- Connecting the thermostat
- Connecting the cell changer
- Connecting the magnetic stirrer
- Optionally, install the aperture baffle

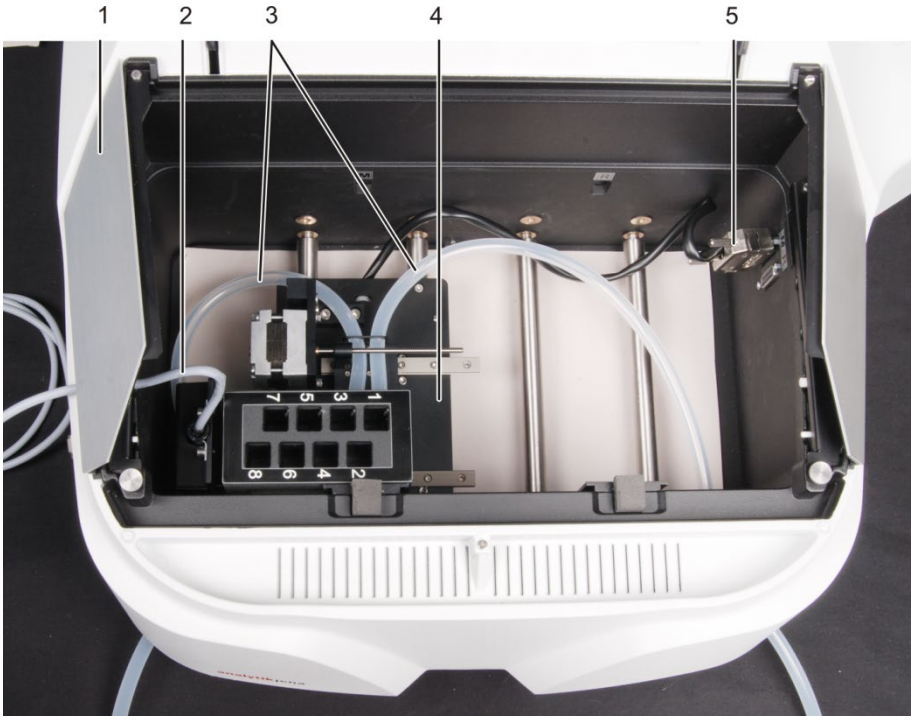



**Fig. 36 8-cell changer in the SPECORD PLUS**

1 Removable side component
2 Connection to the control unit for magnetic stirrer
3 Water hoses
4 Base plate of the cell changer
5 Connector to connect to the SPECORD PLUS

Positioning of support rods

Screw the support rods in the sample chamber into the lower position (→ "Converting the sample chamber" p. 9).

Connecting the thermostat

1. Remove the cover caps from the apertures of the sample chamber in the front panel of the device (1 & 3 in Fig. 4 p. 9).
2. Feed the hoses below the support rods through the apertures to the outside.

   To increase light-proofing the apertures in the sample chamber wall have been provided with steps. Should the hose get stuck at these steps when feeding it through, free it up by rotating it slightly. If it is already visible in the outer aperture, bend it towards the aperture with e.g. a pen.
3. Connect the hoses to the thermostat.

Inserting the cell changer

1. Place the cell changer onto the support rods in the measuring channel and slide it up to the stop against the front sample chamber wall.
2. Press down the cell changer on the right side until it engages on the support rods with a click.
3. Connect the plug to connector (M) in the right sample chamber wall.

Connecting the magnetic stirrer

1. Detach the left side component of the sample chamber (→"Converting the sample chamber" p. 9).
2. Remove one rubber seal from one aperture in the sample chamber wall.

3. Remove the white plug from the seal and insert the cable into the seal.
4. Insert the rubber seal into the aperture with the slot pointing down.
5. Re-attach the side component.
6. Connect the control unit to the mains supply.

Switch on the SPECORD PLUS.

Switch on the SPECORD PLUS and start ASpect UV.

✓ The SPECORD PLUS is automatically initialized: The monochromator moves, and on the monitor the message "Initialization" is shown. The 6-cell changer is automatically detected and displayed in the method parameters.

### 14.1.2 Using aperture baffles

If you are using microcells or semi-microcells made of plastic or quartz or glass cells without blackened rims, install an aperture baffle on the cell changer. The baffle prevents distortion of the measuring result by light that passes through the cell walls or through air bubbles on the cell walls. The installation of the aperture baffle is similar to the installation for the 6-cell changer (→ "Using the aperture baffle" p. 36).

In addition, a baffle can be inserted in the cell duct for cloudy samples (→"Base plate with aperture baffle" p. 130). The baffle separates radiation that is reflected at the cell walls multiple times and therefore is at an oblique angle to the optical axis.

### 14.1.3 Adjusting the 8-cell changer

To position the cells optimally within the beam path the cell changer is adjusted with the aid of a computer.

Adjusting is required in the following situations:

- first use of the cell changer
- after a basic correction
- after transporting the SPECORD PLUS

**Note:** Before adjusting, wait for 2 hours with lamps switched on.

1. Install the empty cell changer in the sample chamber.
2. Switch the SPECORD PLUS on, start the ASpect UV software and wait until the automatic device initialization is completed.
3. Wait for the warm-up time of 2 hours.
4. Measuring using standard cells or semi-microcells: Perform adjustment with empty cell changer.
   Measurements using microcells For adjusting, place a water-filled microcell in the each of the 6 positions.
5. Start the automatic adjustment by choosing INSTRUMENT ▸ ACCESSORY ▸ ADJUSTMENT.

### 14.1.4 Removing the 8-cell changer from the sample chamber

1. To move the cell changer to the parking position: Choose INSTRUMENT ▸ ACCESSORY ▸ SAMPLE POSITION / button: [PARKING].
2. To switch off the accessory: Choose INSTRUMENT ▸ ACCESSORY ▸ ACCESSORY OFF.
3. Disconnect the plug from the connector in the sample chamber wall.
4. Pull up the cell holder on the right side of the base plate until it disengages from the clamp on the support rods with a click.
5. Lift the cell holder out of the sample chamber

Disconnecting the thermostat

1. Disconnect the hoses from the thermostat.
2. Allow the water to drain from the hoses and collect the water.
3. Pull the hoses from the apertures in the front sample chamber wall.
4. Place the cover caps onto the apertures in the front panel of the SPECORD PLUS (1 & 3 in Fig. 4 p. 9) to restore the light-proofing.

Disconnecting the magnetic stirrer

1. Disconnect the stirrer control unit from the mains. Unscrew the cable and the control unit.
2. Unscrew the side component of the sample chamber.
3. Remove the rubber seal from the aperture in the left sample chamber wall and pull the cable of the magnetic stirrer out of the slot of the rubber seal.
4. Close the opening in the rubber seal with the white plug.
5. Insert the rubber seal into the sample chamber aperture with the slot pointing down.
6. Screw on the side component.

## 14.2 Measurement with the 8-cell changer

The measuring parameter configurations and various operating modes are identical for all cell changers available for the SPECORD PLUS. A detailed description is available in chapters "Configuring measuring parameters for the cell changer" p. 49 and "Carrying out measurements with the cell changer" p. 50.

## 14.3 Using two 8-cell changers

In the sample chamber of the SPECORD 200/210/250 PLUS two 8-cell changers can be operated simultaneously, one each in the measuring and reference channels.

Installing two 8-cell changers in the sample chamber

1. Place the support rods in the bottom position of the reference channel. Install the second cell changer analog to the first changer (→ "Installing the 6-cell changer" p. 34).

2. Using the guide rail (5 in Fig. 34) (included), align both cell changers with respect to each other so that they have the same distance from the front sample chamber. Correct the adjustment of the stop bolts (7 in Fig. 35 p. 41), if required.
3. In case of temperature control by an external thermostat connect the two cell changers with a short piece of hose. Feed the water connection hoses of the cell changers to the outside and connect them to the thermostat.
4. Connect the electrical connection cable for the magnetic stirrer as described above.
   Feed the connection of the cell changer in the reference beam path to the outside through the right sample chamber side. Connect both magnetic stirrers to the control unit using the Y adapter supplied.
5. Adjust the two 8-cell changers in the beam path analog one 8-cell changer (→ "Adjusting the 8-cell changer" p. 44).
6. In order to use the adjustment for subsequent measurements, use the adhesive labels (supplied). Attach the "M" and "R" labels to the step motors of the cell changers in the measuring and reference beam paths, making sure they clearly visible.

Measurements with two 8-cell changers

The configuration for the use of two cell changers is found in chapter "Carrying out measurements with two cell changers" p. 53.

# 15 Cell carousel

The cell carousel is an automatic sample changing system for 15 cells. The cell carousel is suitable for the following cells:

| | |
|---|---|
| Pathlength | 10 mm |
| Cell width | 12.5 mm |
| Minimum filling height | 20 mm |
| Radiation height | 5 – 15 mm |

Layout

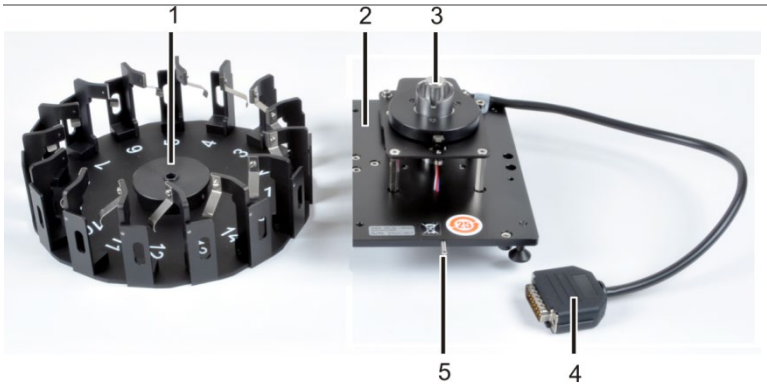



**Fig. 37 Layout of the cell carousel**

1 Cell plate
2 Base plate with drive
3 Drive unit
4 Interface connection to the SPECORD PLUS
5 Contact pin

Installing the cell carousel

1. Screw the support rods in the sample chamber into the lower position (→ "Converting the sample chamber" p. 9).
2. Place the base plate onto the left sample chamber rod in the measuring channel and slide to the front sample chamber wall until the contact pin touches the sample chamber wall.
3. Press down the base plate of the sample holder until it engages on the support rods with a click.
4. Connect the cell carousel to the connection (**M**) in the sample chamber. The cable must not protrude into the beam path.

**Fig. 38 Base plate of the cell carousel installed in the SPECORD PLUS**

5. Place the sample plate onto the drive unit. Rotate the sample plate until it engages in the groove.

6. Screw the sample plate onto the drive unit using the knurled head screw supplied.
   ✓ The cell carousel is now fully mounted and ready for measurements.


**Fig. 39 Cell carousel fully mounted**

Removing the cell carousel from the sample chamber

1. To switch off the accessory: Choose INSTRUMENT ▸ ACCESSORY ▸ ACCESSORY OFF.
2. Disconnect the plug from the connector in the sample chamber wall.
3. Unscrew the sample plate from the drive unit.
4. Pull up the drive unit on the right side of the base plate until it disengages from the clamp on the support rods with a click.
5. Lift the drive unit out.

Measurements with the cell carousel

The measuring parameter configurations and various operating modes are identical for all cell changers available for the SPECORD PLUS as well as for the cell carousel. A detailed description is available in chapters "Configuring measuring parameters for the cell changer" p. 49 and "Carrying out measurements with the cell changer" p. 50.

# 16 Measurement with the cell changer

## 16.1 Configuring measuring parameters for the cell changer

The cell changers are automatically detected during the device initialization in ASpect UV and displayed in the method parameters. In ASpect UV, the following method parameters are available in all modules:

GENERAL tab, MULTIPLE MEASUREMENTS group

| Parameter | Description | |
|---|---|---|
| NUMBER | Number of multiple determinations | |
| CYCLIC FOR | SAMPLE | All repeat measurements are made with one sample. Then the next sample is indexed and the measurements are performed there. |
| | Batch | At first, each sample in the cell changer is measured once. Then the cell changer goes back to position 1 and starts the next measurement of each sample. |
| TIME CONTROLLED | If enabled, the repeat measurements take place automatically after the INTERVAL TIME entered. | |

**Note**: In the PHOTOMETRY module, the average value and the standard deviation of the measured values of multipoint detections are displayed in the sample table. The results of the individual measurements are listed adjacent in a separate table.

ACCESSORIES tab

| Parameter | Description |
|---|---|
| MANUAL START | Each measurement of a sample in the cell changer is started manually by pressing a button. |
| SYNCHRONOUS | Only if two cell changers are used<br>**Enabled:** The two cell changers move **synchronously**. A separate reference can be used for each sample.<br>**Disabled:** Both cell changers are moved consecutively through the beam paths (**offset** mode). In this way, both cell changers are used for the sample measurement. At first, the samples in the reference channel are measured followed by the samples in the measuring channel. In offset mode, the last cell position of the cell changer in the reference channel and the first cell position of the cell changer in the measuring channel are used to record the reference beam. They must remain empty or be populated with reference samples. |

Move to sample position

Via the menu item INSTRUMENT ▸ ACCESSORY ▸ SAMPLE POSITION of the main window, the cell changer can be moved to specific positions and adjusted:

| Button | Description |
|---|---|

| | |
|---|---|
| [POS.1] | Move the cell holder to position 1. |
| [POS. +1] | Move the cell holder to the next position. |
| [ADJUSTMENT] | For 6- and 8-cell changers<br>Start adjustment (→ "Adjusting the 6-cell changer" p. 77) |
| [PARKING] | Move the cell changer into the "parking position" where the cell block is centered above the base plate.<br>In this position the cell changer can be easily installed into and removed from the SPECORD PLUS as well as packaged. |
| **Synchronous** | Adjustment of the operation of 2 cell changers (see above) |

## 16.2 Carrying out measurements with the cell changer

Preparing the sample sequence

ASpect UV automatically creates a sequence with as many rows as the number of spaces available in the cell changer.

In each sequence the sample type, name and other individual sample data, such as the concentration of standards, are stored. After starting the measurement the sequence is automatically processed and the results are displayed in the results window. See operating instructions "ASpect UV – Software for UV/Vis Spectrometer" for further information about sample types.

The following sample types can be selected via the buttons in the MODE OF SAMPLE TYPE group:

| Parameter | Description |
|---|---|
| REFERENCE [R] | Reference measurement |
| BLANK [0] | Blank value measurement |
| SAMPLE [S] | Sample measurement |
| NO MEASUREMENT [NO] | The corresponding measuring space in the accessory is not filled. No measurement is performed. |
| STANDARD [STD] | Standard measurement for calibration<br>**Note:** The [STD] button is not displayed until the CALIBRATION option on the CALIBRATION tab has been activated. |

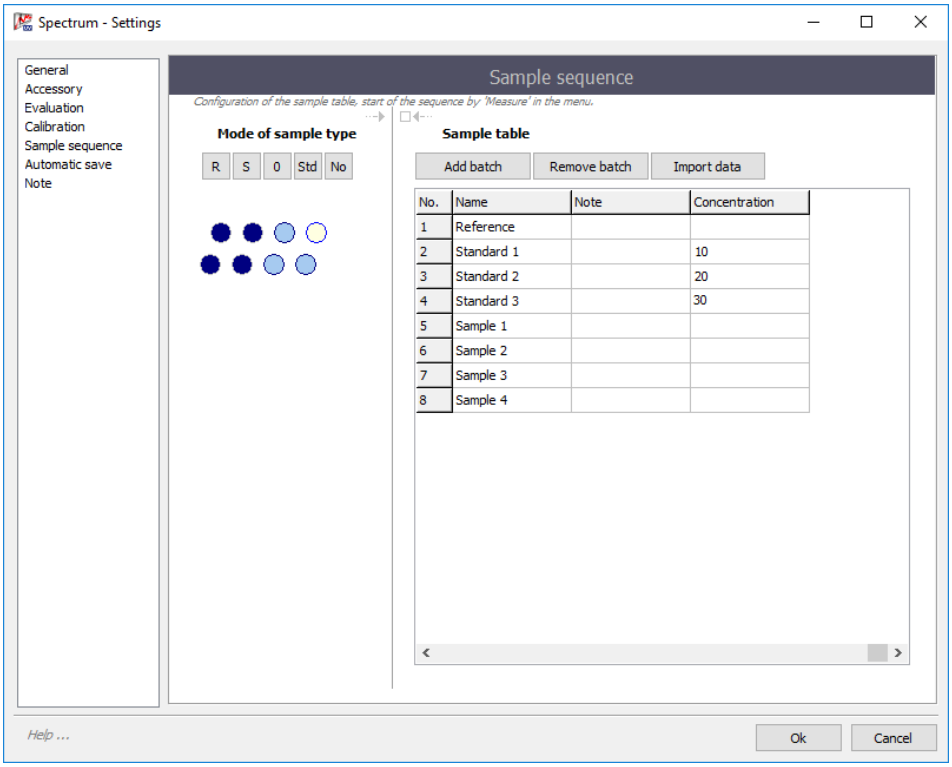



**Fig. 40 Sample sequence with different sample types**

If more samples are to be measured in a sequence than the number of spaces available in the cell changer, the sample table can be extended by additional batches (loads) via the [ADD BATCH] button. [REMOVE BATCH] can be used to remove these batches.

Separate measuring of the reference sample

A separate sample measurement is required if there is no reference in first position of the sample sequence and no reference matching the current method parameters is available from a previous series of measurements

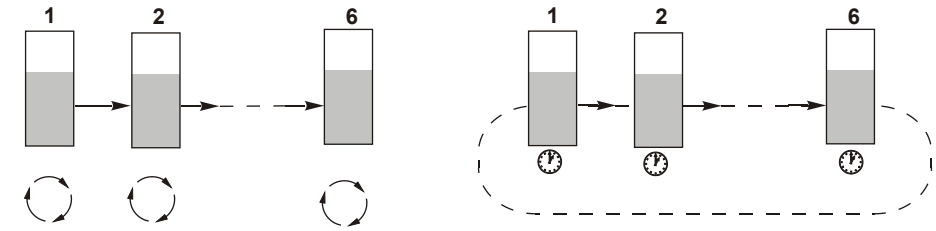
1. Start the reference measurement with [REFERENCE] in the menu and function bar of the document window, and select a position for the reference measurement.
2. Place a reference in the selected cell position or leave the position empty. If required, also fit the reference beam path with a reference (→ "Measuring the reference sample" p. 11).
3. After measuring the reference, fill the cell changer in accordance with the entries in the sequence.
4. Start the processing of the sequence with [MEASURE].

Measurement with reference sample in the cell changer

1. In the sequence, set the reference measurement in first position.
2. Fill the cell changer with reference and samples in accordance with the entries in the sequence.
3. Start the processing of the sequence with [MEASURE] in the menu and function bar of the document window.

Kinetic measurements with the cell changer

In ASpect UV it is possible to perform kinetic measurements in the KINETICS module. The cell change takes place in two different modes adapted to the reaction speed: SAMPLE and CHARGE modes.




SAMPLE mode for fast reaction kinetics

BATCH mode for slow reaction kinetics

**Fig. 41 Modes for the cell change during kinetic measurements**

SAMPLE mode for fast reaction kinetics

For **fast kinetics** the complete reaction in a cell is tracked. This is followed by a change to the next cell and the reaction start in this cell. Each cell is processed in this way.

The measuring start in each cell takes place via a separate start command (MANUAL START option) to have enough time to add the start reagents to the sample.

BATCH mode for slow reaction kinetics

For **slow kinetic reactions** a measurement is carried out in each cell as part of a cycle. During the next cycle the measurements are repeated in each cell, and so on until the end of the total measuring time.

For both measuring modes, the following method parameters must be set on the GENERAL tab in the TIME CYCLE group:

| Parameter | Configuration |
|---|---|
| MEASURING TIME | Total measuring time |
| INTERVAL TIME | Time between the recording of two consecutive measurement values |
| DELAY TIME | Time between the start of measuring and the actual start of the first measurement. |
| DISPLAY TIME AS | Time unit for the x-axis of the graphical representation |
| CYCLIC | Choice between the SAMPLE and BATCH measuring modes |

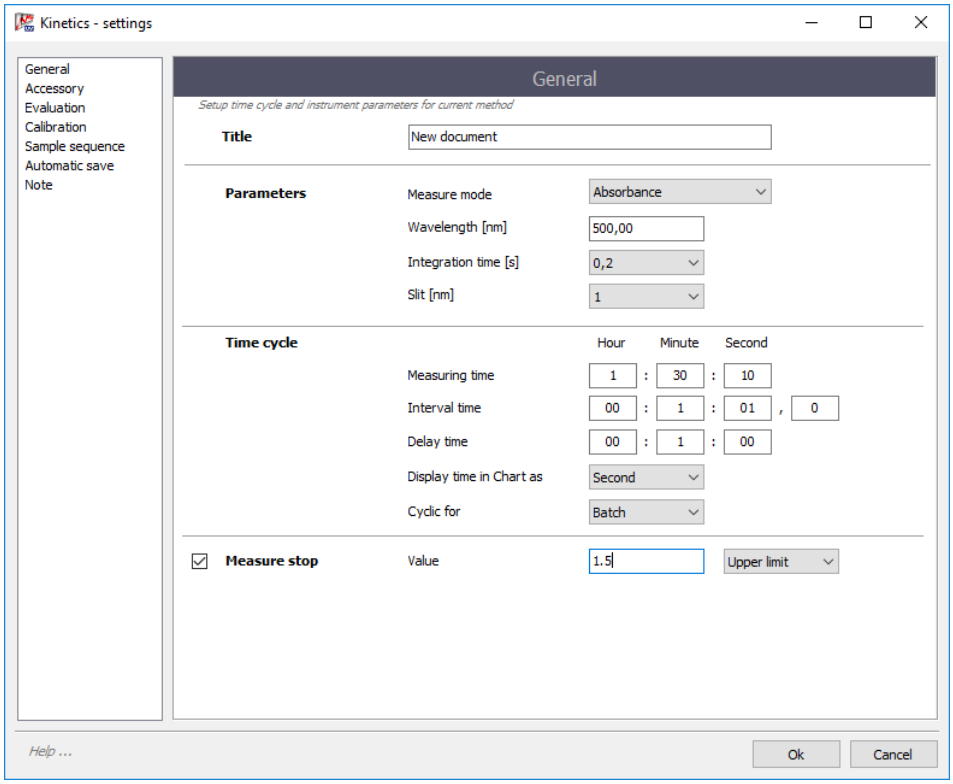



**Fig. 42 Exemplary settings for slow reaction kinetics**

Automatic measurement cancellation can be specified for kinetic measurements once the absorption or transmission measurement parameters have reached a lower and/or upper limit.

## 16.3 Carrying out measurements with two cell changers

The main functions for measurements with two 6- or 8-cell changers are similar to the software setting for one cell changer (→ see section"Configuring measuring parameters for the cell changer" p. 49).

Offset cell changer mode

In offset mode, both cell changers are moved consecutively through the beam paths so that both cell changers are used for sample measurement.

The maximum number of samples that can be measured is 10 if two 6-cell changers are used, and 14 if two 8-cell changers are used.

For **offset operation**, the SYNCHRONOUS option must be deactivated in the method settings on the ACCESSORY tab. The sample table is automatically adapted to this option.

The cell positions are populated from right to left **starting with the first position** in the cell changer **in the reference channel**.

The **last cell position** of the cell changer **in the reference channel** and the **first cell position** of the cell changer **in the measuring channel** are used in offset mode to record the reference beam. They must remain empty or be populated with reference samples.

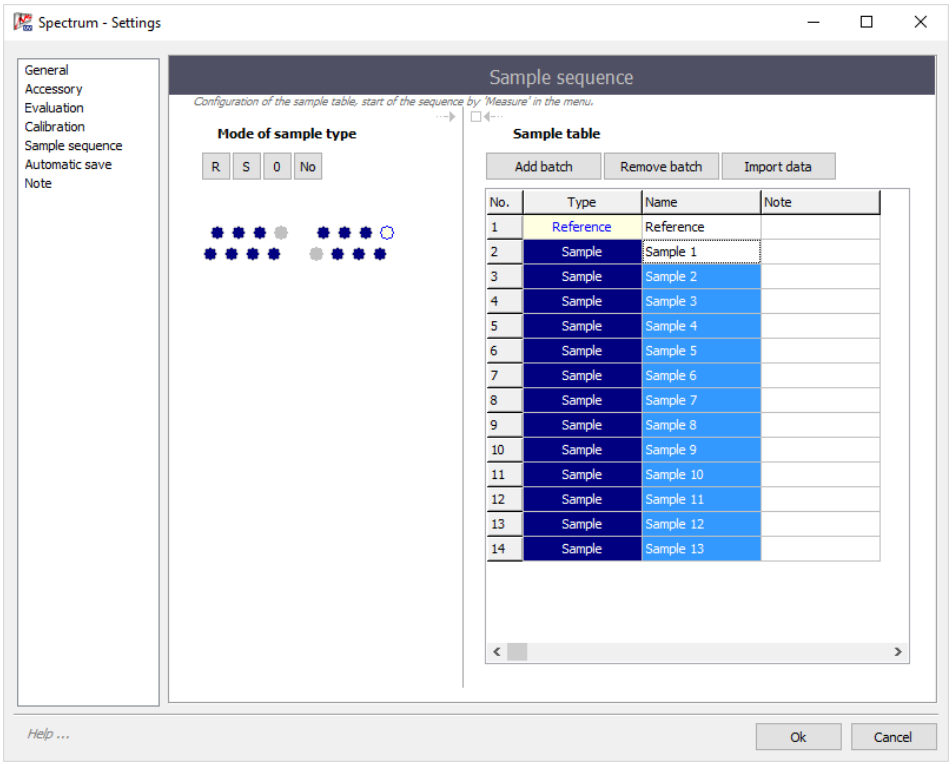



**Fig. 43 Sample sequence for two 6-cell changers**

Reference and sample measurement in offset mode

In offset mode, the reference can be considered in two different ways:

- The reference measurement is carried out separately before the sequence is started. The selected cell position remains empty or is filled with a reference sample.
- The reference measurement takes place as part of the sequence. The first position of the cell changer in the reference beam path is filled with the reference or remains empty.

The options for measuring the reference are described in section "Measuring the reference sample" p. 11.

Synchronous cell changer mode

In **synchronous mode**, an individual reference sample is carried along in the reference beam path for each sample in the measuring channel. The samples are positioned in the cell changer of the measuring channel and the references for each sample are positioned in the cell changer of the reference channel.

For synchronous operation, the SYNCHRONOUS option must be activated in the method settings on the ACCESSORY tab. A sample sequence with 6 or 8 rows, respectively, is created. The reference samples are not represented in the sequence.

Carrying out the measurement in synchronous operation

1. Carry out a reference measurement with empty cell changers.
2. Fill the cell changers. Place the samples into the cell changer in the measuring channel (**M**) and the reference samples into the corresponding positions of the cell changer in the reference channel (**R**).

# 17 Peltier-tempered accessories

## 17.1 General safety instructions for Peltier-tempered accessories

The following safety instructions apply to all Peltier-tempered accessories:

### WARNING

**Electric shock at the temperature control unit and heat exchanger!**

Disconnect the mains plug before opening the device or removing the covers! Components carrying mains or high voltages may be exposed.

The mains plug must only be inserted into a shock-proof socket to guarantee protection class I (protective conductor connection) for the device. The protective effect must not be invalidated by the use of an extension line which does not have a protective conductor.

Only fuses of the type specified must be used.

### CAUTION

**Risk of burns due to hot surfaces**

After operation at high temperatures, allows the cells to cool down sufficiently before exchanging.

### Attention

**Possible overheating due to heat build-up**

The ventilation slits of temperature control unit and heat exchanger must remain unobstructed!

**Possible short-circuit caused by liquids**

Make sure that no liquids enter into the device. The following has to be observed:

- Do not place glasses or any other vessels containing liquids on the devices.
- When working with a watery system at temperatures below freezing there is a risk of destroying the cells due to the expansion of the ice.
- Place the control unit **on top of** the heat exchanger, or place both devices next to each other.

## 17.2 Peltier-tempered cell holder, air-cooled

The Peltier-tempered cell holder allows the temperature control of cells with the dimensions 12.5, x 12.5 x 45 - 46 (L x W x H in mm) and 10 mm pathlength. Commercially available 10-mm macro cells made of glass, quartz or plastic as well as macro and semi-microcells with cylindrical stirring bottom can be used. Owing to the power limits, only cells with a flat bottom and a bottom thickness of no more than 1.5 mm are suited.

The temperature control of the cell holder is carried out via a separate control unit. The rear of the Peltier element is air-cooled via a heat exchanger with cooling ribs. The required air is aspired and expelled via light-proof ducts.

As control sensor a sensor is used and positioned at the bottom outer corner of the cell block. The cell holder further also features two additional sensors for the optional registration of the holder or cell temperature.

The cell sensor is designed specifically for standard cells with round plugs from PTFE and can remain in the cell during the optical measurement.

The cell holder is equipped with a magnetic stirrer by default so that an even temperature distribution in the cell is achieved more quickly. However, the stirring power is not sufficient to homogeneously distribute e.g. reagents in the sample. The stirring speed is set at the temperature control unit. Optimum stirring of the sample is achieved when using magnetic stirring rods with a diameter of 2 mm and a length of 5 mm. For cells with cylindrical stirring bottom, it is also possible to use magnetic stirring rods with a diameter of 3 mm and a length of 6 mm. The stirring power must be increased gradually in order to avoid jamming of the magnetic stirrer.

Two cell holders can be used in the measuring and the reference channels. A separate temperature control unit is required for each cell holder.

Standards

The Peltier-tempered cell holder has been constructed and tested in accordance with the following standards and guidelines:

- DIN EN 61010-1 (IEC 1010-1)
- DIN EN 55011 class B
- DIN EN 61326-1
- DIN EN 61000-3-2
- DIN EN 61000-3-3

Safety instructions

Observe the General Safety Instruction for the operation of the Peltier-tempered accessories (→ "General safety instructions for Peltier-tempered accessories" p. 55).

### 17.2.1 Technical data and layout

| | |
|---|---|
| Principle | Thermoelectric heating and cooling |
| TEC rear cooling | air-cooled |
| Guaranteed controlled temperature range at 25 °C ambient temperature | +10...+60 °C for design with heat exchanger type A<br>-5...+105 °C for design with heat exchanger type B |
| Preset range for the block temperature | -20 ... +105 °C |
| Preset accuracy | 0.1 °C |
| Display accuracy | 0.1 °C |
| Control accuracy | +/- 0.1 °C |

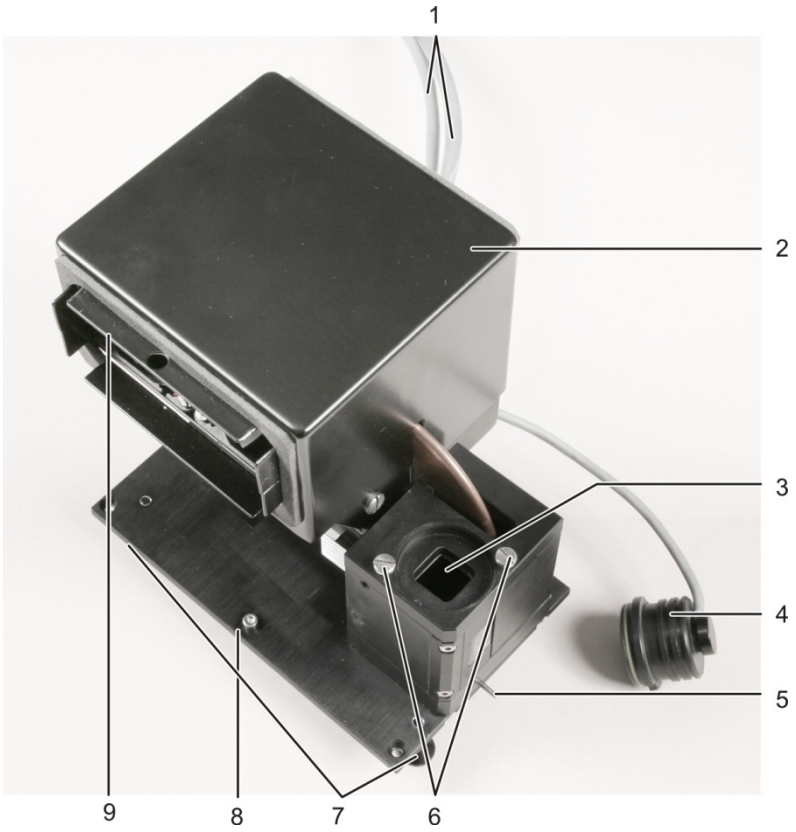
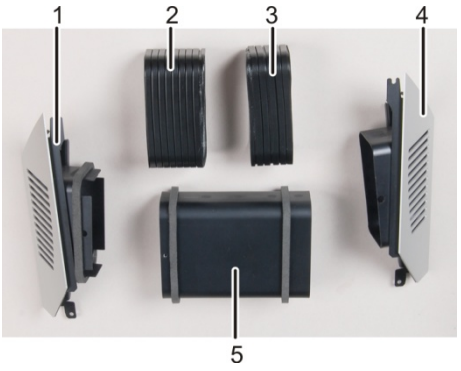



**Fig. 44 Peltier-tempered cell holder, air-cooled**

1 Connection cables to the temperature control unit
2 Heat exchanger housing
3 Cell block with quartz windows
4 Cover cap
5 Contact pin
6 Locking screws
7 Contact feet for attachment to the support rods
8 Base plate
9 Recesses to attach the air ducts




1 left side wall with air vents
2 flexible longer air duct
3 flexible shorter air duct
4 right side wall with air vents
5 fixed air duct

**Fig. 45 Air ducts for Peltier-tempered cell holder**

## 17.2.2 Installing the cell holder in the sample chamber

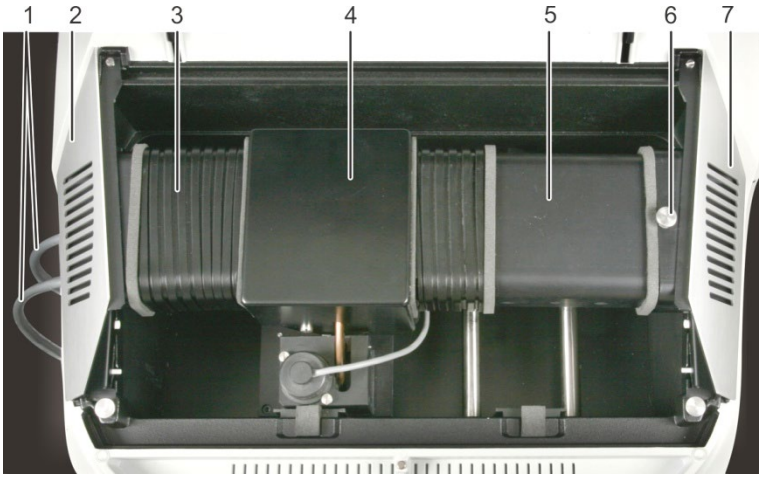
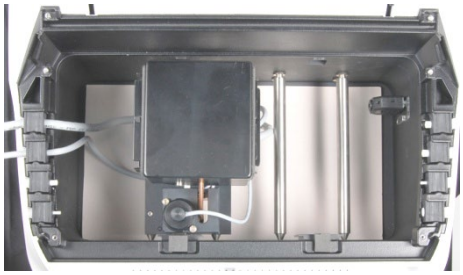



**Fig. 46 Installing the Peltier-tempered cell holder (air-cooled) in the sample chamber**

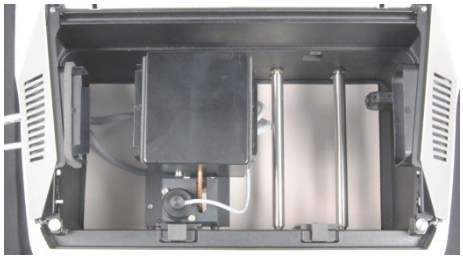
1 Connection cables to the temperature control unit
2 Left side component
3 Longer flexible air duct
4 Cell holder
5 Fixed air duct
6 Knurled head screw
7 Right side component

The installation of the cell holder is the same for all devices in the SPECORD PLUS family. In the SPECORD 50 PLUS, the lengths of the fixed and flexible air ducts are adapted to the center position of the measuring channel in the sample chamber.

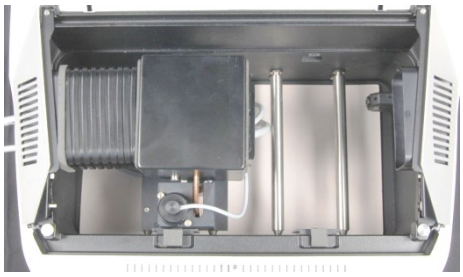
1. Screw the support rods in the sample chamber into the top position (→ "Converting the sample chamber" p. 9).
2. Connect the plug to connector (**M**) in the right sample chamber wall.
3. Unscrew the side components of the sample chamber walls.
4. Lay the connection cables below the support rods to prevent them protruding into the beam path later.



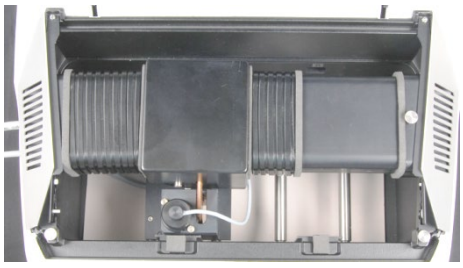
5. Place the cell holder onto the support rods in the measuring channel (**M**). The cell block points forward.
6. Slide the cell holder up to the stop against the front sample chamber wall. Press down the base plate on the right side until the cell holder engages with a click.
7. Remove the two rubbers seals with the larger plugs from the apertures in the left side wall. This is easier if you tilt the seals.



8. Remove the white plugs from the seals and press the electrical cable connections for the heat exchanger and control unit into the slots.
9. Insert the rubber seals into the apertures with the slots pointing down.
10. Screw on the side walls with the air vents.



11. Mount the side wall and the cell holder with the longer flexible air duct. Pull the lamellae apart until the metal hose rests both on the adapters at the heat exchanger housing and the side wall.



12. Place the fixed air duct onto the adapters of the right sample chamber wall and fasten it using the knurled head screw.
13. Connect the cell holder and the fixed air duct with the shorter flexible air duct.
14. Connect the connection cables to the temperature control unit (→ "Temperature control unit for Peltier-tempered accessories" p. 82).
15. Place the cell into the cell block and close it with the cover cap. Lock the cover cap by turning it slightly.

    ✓ The cell holder is fully installed.

### 17.2.3 Using the cell sensor

The software can be used to capture either the temperature in the cell block or (using the cell sensor) in the cell.

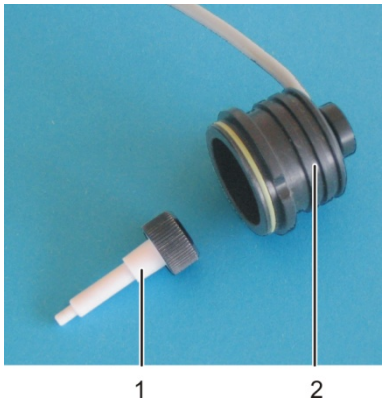
**Attention**

**Fragile sensor**

Do not use force when attaching the sensor. It is not necessary to press the sensor in place because it receives enough pressure from the contact pins in the cover cap.

**Flashing temperature display**

If the temperature display (Fig. 65 p. 84) in the control unit is flashing, either the cell sensor is not properly connected to the connection socket or the sensor is faulty.




1 Cell sensor
2 Cover cap of the cell holder

**Fig. 47 Cell sensor**

1. Seal a standard cell with round plug with the cell sensor supplied.
2. Place the cell into the cell block and close it with the cover cap (the grooves are opposite the locking screws).
3. Lock the cover cap by turning it slightly.
4. Turn the change-over switch at the control unit to "cell".

Extending the immersion depth of the sensor

The contact cap is equipped with a set screw in the cover cap. By loosening this screw it is possible to vary the immersion depth of the contact pins in the cover cap by a maximum of 4 mm. This permits the use of the cell sensor with non-standardized cells.

Sensors for ultra-microcells

A special sensor is available for ultra-microcells. For this purpose, the cover cap must be replaced. A conversion kit, consisting of a screwdriver, cover cap and cell sensor, is available to order.

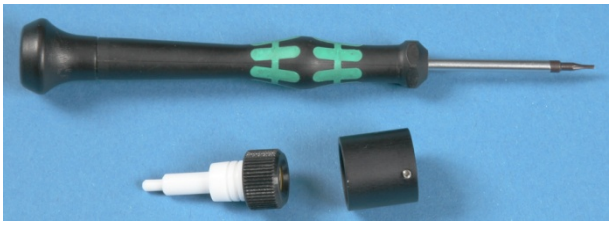
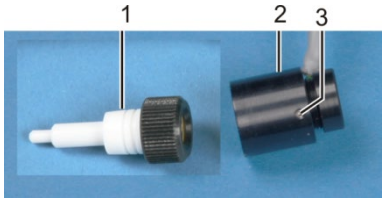


**Fig. 48 Conversion kit for ultra-microcells**

1. Using the screwdriver loosen the set screw in the cover cap.
2. Pull the cover cap for the standard cell off the contact cap.
3. Slide the cover cap for ultra-microcells onto the contact cap and tighten the set screw.




1 Cell sensor for ultra-microcells
2 Cover cap
3 Set screw

**Fig. 49 Cover cap and cell sensor for ultra-microcells**

### 17.2.4 Temperature control for Peltier-tempered cell holders

The temperature control takes place via the ASpect UV software. After the temperature control unit is switched on, the temperature to which the unit is set is established as the start temperature (→ see section "Temperature control unit for Peltier-tempered accessories" p. 82). After starting the measurement all subsequent temperature control is based on the measuring parameters configured in ASpect UV.

The Peltier-tempered cell holder is detected during the device initialization and displayed in the method parameters.

The actual temperatures of the cell and of the cell block are displayed in the ASpect UV software via INSTRUMENT ▸ ACCESSORY ▸ TEMPERATURE. The possible specification range for the block temperature of the accessory is also displayed here.

With Peltier-tempered accessories, the following temperature operation modes are possible:

| Mode | Description |
|---|---|
| NONE | The temperature mode NONE is available in the modules Photometry, Spectrum and Kinetics.<br>In this mode, the temperature of the cell holder/changer is not controlled via the ASpect UV program. |
| FIXED TEMPERATURE | Activation of a constant temperature<br>The measurement is taken when the target temperature is reached. The temperature mode CONSTANT is available in the modules Photometry, Spectrum and Kinetics. |

| | |
|---|---|
| VARIABLE | Activation of defined temperature values (max. 200).<br>The measurement is taken when the temperature is reached. The temperature mode VARIABLE is available in the modules Photometry and Spectrum. |

The temperature is optionally sensed either by the sensors in the cell block or by the cell sensor.

Temperature mode: FIXED TEMPERATURE

To activate and hold a constant temperature, select the FIXED TEMPERATURE mode and make the following settings on the ACCESSORY tab:

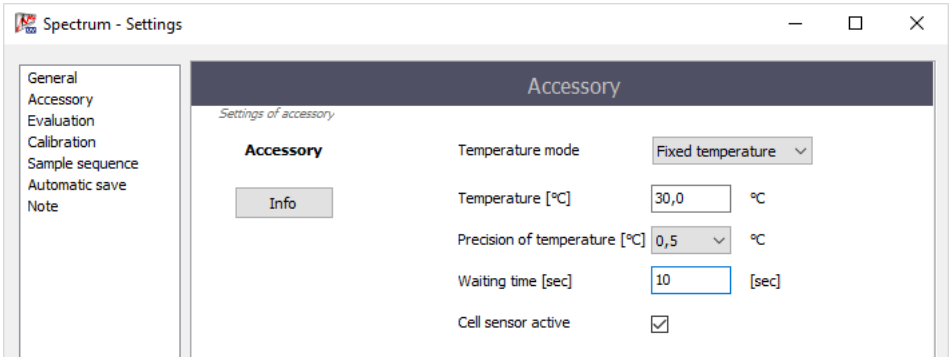



**Fig. 50 Measuring parameter configuration for Peltier-tempered cell holder – temperature mode: constant**

| Parameter / button | Description |
|---|---|
| TEMPERATURE MODE | FIXED TEMPERATURE – Set a constant temperature. |
| TEMPERATURE [°C] | Enter the target temperature (for the cell block). |
| PRECISION OF TEMPERATURE | Select the accuracy of the temperature control.<br>Range: 0.1°C ... 2°C |
| WAITING TIME | Enter the waiting time from reaching the target temperature to starting the optical measurement. |
| CELL SENSOR ACTIVE | When the target temperature is reached, the registered cell temperature is recorded. The measured value (A / %T) is allocated to the cell temperature. This allows a more exact temperature measurement.<br>If the option is disabled, the block temperature (standard temperature) is indicated as an abscissa value. |
| [INFO] | Display of the current temperatures in cell and cell block, display of the target temperature range. |

To enable the cell sensor, the change-over switch at the temperature control unit must be switched to "cell" in addition to the software settings (→ "Using the cell sensor" p. 60).

Temperature mode: VARIABLE

In VARIABLE temperature mode you can activate up to 200 temperature values. The measurement is taken when the target temperature is reached. Make the following settings under Method on the ACCESSORY tab:

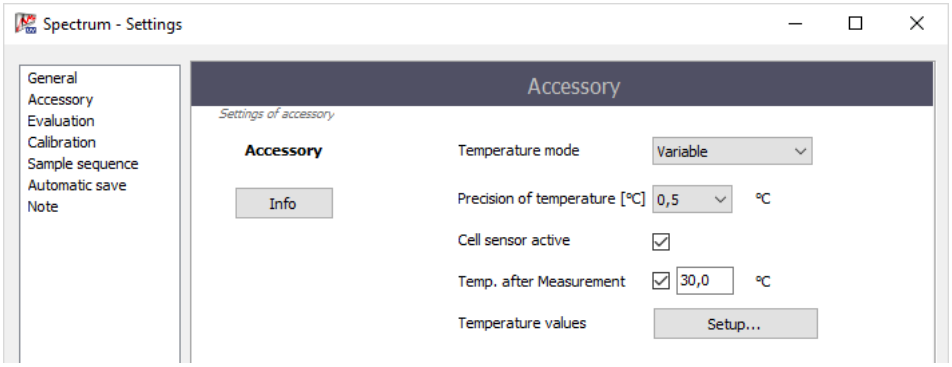



**Fig. 51 Measuring parameter configuration for Peltier-tempered cell holder – Temperature control: variable**

| Parameter / button | Description |
|---|---|
| TEMPERATURE MODE | VARIABLE– Select various temperature values that are set one after the other during the measuring phase. |
| PRECISION OF TEMPERATURE | Select the accuracy of the temperature control.<br>Range: 0.1°C ... 2°C |
| CELL SENSOR ACTIVE | When the target temperature is reached, the registered cell temperature is recorded. The measured value (A / %T) is allocated to the cell temperature. This allows a more exact temperature measurement.<br>If the option is disabled, the block temperature (standard temperature) is indicated as an abscissa value. |
| TEMP. AFTER MEASUREMENT | If enabled, the set temperature is established and held after the last measurement. |
| TEMPERATURE VALUES | Use [SETUP] to open the window for entering the temperature values. |
| [INFO] | Display of the current temperatures in cell and cell block, display of the target temperature range. |

VARIABLE TEMPERATURE MODE window

After clicking on [SETUP] the window VARIABLE TEMPERATURE MODE appears. The parameters of the temperature stages can be edited individually in the table rows or defined automatically using the checkboxes and input fields.

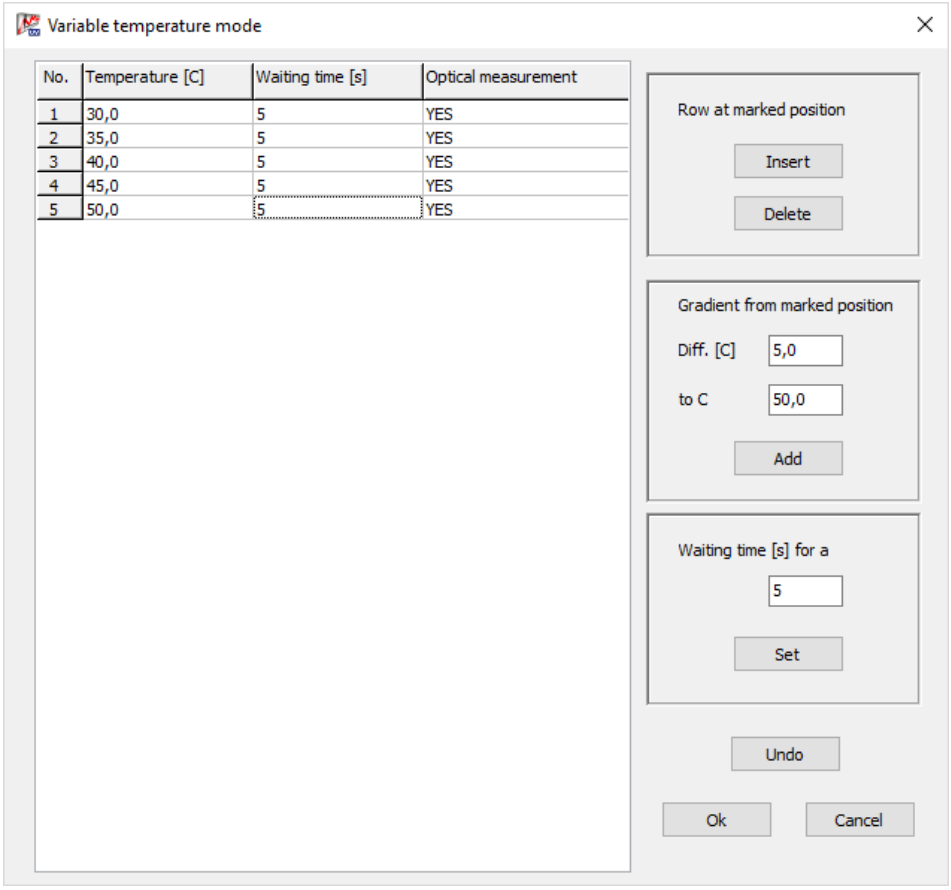



**Fig. 52 VARIABLE TEMPERATURE MODE window for Peltier-tempered cell holders**

The table in the VARIABLE TEMPERATURE MODE window contains the TEMPERATURES to be established, the WAITING TIME between reaching the temperature and the start of the measurement, and the option to start or not to start a measurement at the respective temperature stage (OPTICAL MEASUREMENT).

The parameters of the temperature stages can be edited individually in the table rows or defined automatically using the checkboxes and input fields.

| Parameter / button | Description | |
|---|---|---|
| [INSERT] | Inserts an additional cell (additional temperature range) at the selected location of the table. | |
| [DELETE] | Deletes a selected table cell. | |
| GRADIENT FROM MARKED POSITION | Automatically generate a temperature gradient from a highlighted table row. Starting with the start temperature of the highlighted table row further temperature stages are inserted into the table in steps until the final temperature has been reached. | |
| | DIFF. [°C] | Temperature difference of a temperature stage |
| | TO °C | Final temperature of the gradient |
| | [ADD] | Add temperature gradient at the highlighted position |

| | |
|---|---|
| WAITING TIME FOR ALL [S] | Waiting time from reaching the target temperature to starting the optical measurement<br>With [SET] the temperature entered is transferred to all rows of the table. |
| [UNDO] | Reverse the last action. |

## 17.2.5 Operation with sample chamber flushing

Depending on the room temperature and the relative humidity, the water of the ambient air condensates below a certain block or cell temperature (dew point) on the cell walls and the cell block. This distorts the measuring results.

The following context applies for the start of condensate formation (dew point temperature):

$$\vartheta_K = \left(\frac{\text{relativeLuftfeuchte}}{100}\right)^{0,1247} * (109,8 + \vartheta_R) - 109,8$$

$\vartheta_K$ - dew point temperature in °C

$\vartheta_R$ - room temperature in °C

For a room temperature of 20 °C and a relative humidity of 60 % the dew point temperature is 12 °C. The condensate formation can be avoided by flushing the sample chamber with dry gas.

1. Feed an as thick as possible black hose with an internal diameter of at least 6 mm through one of the apertures of the sample chamber. Close the aperture so that it is light-proof.
2. Place the muzzle of the hose at the center of the front sample chamber wall. To avoid unstable temperatures the gas should not be blown directly onto the cell holder.
3. Flush the sample chamber with 800 – 1000 l/h dry gas, e.g. air, nitrogen or argon.

**Note:** Before working at low temperatures, the sample chamber should be pre-dried. Clean out the sample chamber by operating the Peltier cell holder/changer at +80 °C for approx. 10 minutes.
At low temperatures avoid opening the sample chamber for unnecessary long periods of time to prevent humid room air from entering.

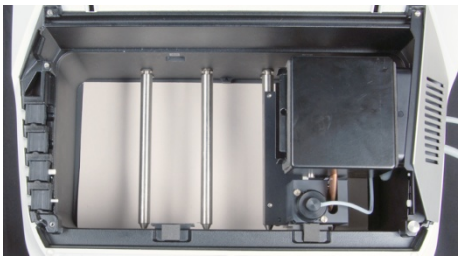
## 17.2.6 Using two Peltier-tempered cell holders

Installing the cell holder in the sample chamber

The SPECORD 200/210/250 PLUS can accommodate two cell holders in the sample chamber. The installation of the two cell holders takes place from the right to the left side of the sample chamber.

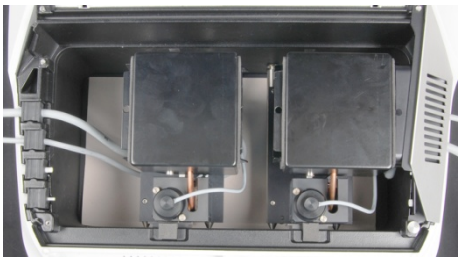
1. Screw the support rods in the sample chamber into the top position (→ "Converting the sample chamber" p. 9).
2. Plug one identification connector each into the connections (**M**) and (**R**) in the right sample chamber wall.
3. Unscrew the side components.

4. Place the cell changer onto the support rods.
5. Remove the two rubbers seals with the larger plugs from the apertures in the right side wall.
6. Remove the white plugs from the seals and press the electrical cable connections for the heat exchanger and control unit into the slots.
7. Insert the rubber seals into the apertures with the slots pointing down.

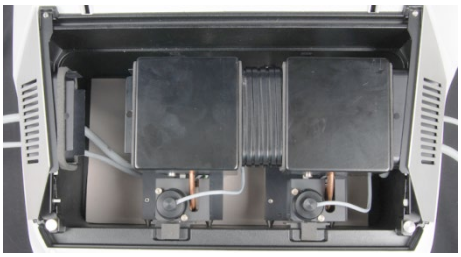


8. Insert the right side wall with the air vents. Do not screw on yet.
9. Place the cell holder onto the support rods in the reference channel (**R**). The cell block points forward.
10. Slide the cell holder up to the stop against the front sample chamber wall. Press down the base plate on the right side until the cell holder engages with a click.

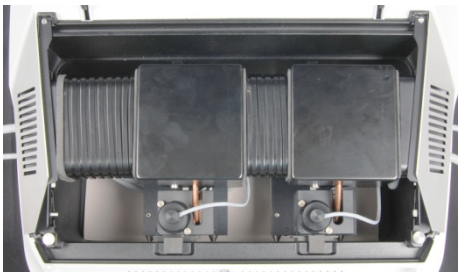
    Slide the adapters of the cell holder into the adapter of the air vents on the right side wall- The connection is now light-proofed.
11. Screw on the side component.



12. Lay the connection cables of the second cell holder below the support rods to prevent them protruding into the beam path later.
13. Mount the second cell holder in the measuring channel (**M**).
14. Lay the connection cables through the apertures in the left side wall.



15. Screw on the left side wall.
16. Connect the two the cell holders to the shorter flexible air duct. Pull apart the lamellae until both ends of the air duct sit on the adapters of the heat exchangers.



17. Connect the left side wall and the heat exchanger of the cell holder in the measuring channel (**M**) to the longer flexible air duct.
18. Connect the connection cables to the temperature control units (→ see section "Temperature control unit for Peltier-tempered accessories" p. 82). You need one control unit for each cell holder.
19. Place the cells into the cell blocks and close with the cover caps. Attach the cover caps by turning them slightly.

Measuring with two cell holders

The measurement with two Peltier-tempered cell holders is carried out as in section "Temperature control for Peltier-tempered cell holders" p. 61. The same temperatures are set in both cell holders.

### 17.2.7 Care

The Peltier-tempered cell holder is largely maintenance-free.

- Use care when handling the sample substances to avoid contamination, especially inside the cell block.
- Wipe spilled samples or reagents immediately with an absorbent cloth or piece of paper.
- If in spite of all care contamination (sample substances etc.) occurs, the interior of the cell block can be rinsed with ethanol or water with added dishwashing detergent. To drain the rinsing liquid there is an M4 screw with hexagon socket at the bottom of the base plate.
- Store the Peltier-tempered cell holder in the packaging supplied.

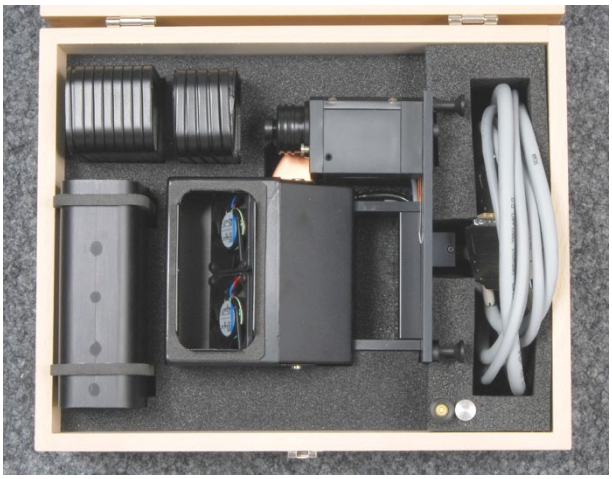


**Fig. 53 Peltier-tempered cell holder in its packaging**

## 17.3 Peltier-tempered cell holder with external heat exchanger

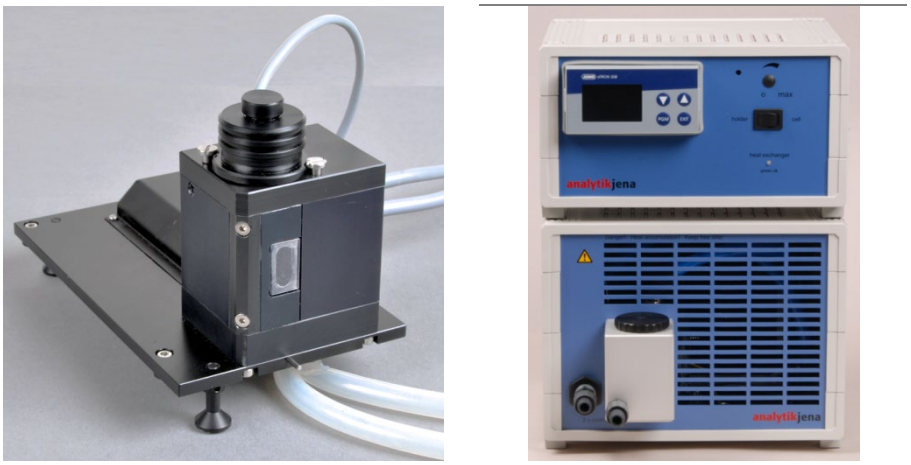



**Fig. 54 Peltier-tempered cell holder with temperature control unit and heat exchanger**

The Peltier-tempered cell holder allows the temperature control of cells with the dimensions 12.5, x 12.5 x 45 - 46 (L x W x H in mm) and 10 mm pathlength. Commercially available 10-mm macro cells made of glass, quartz or plastic as well as macro and semi-microcells with cylindrical stirring bottom can be used. Owing to the power limits, only cells with a flat bottom and a bottom thickness of no more than 1.5 mm are suited.

The temperature control of the cell holder is carried out via a separate control unit. As control sensor a sensor is used and positioned at the bottom outer corner of the cell block. In addition to the control sensor the cell holder features two further sensors for the optional registration of the block or cell temperature.

The recooling of the Peltier element is via a heat exchanger.

The cell sensor is designed specifically for standard cells with round plugs from PTFE and can remain in the cell during the optical measurement.

The cell holder is equipped with a magnetic stirrer by default so that an even temperature distribution in the cell is achieved more quickly. However, the stirring power is not sufficient to homogeneously distribute e.g. reagents in the sample. The stirring speed is set at the temperature control unit. Optimum stirring of the sample is achieved when using magnetic stirring rods with a diameter of 2 mm and a length of 5 mm. For cells with cylindrical stirring bottom, it is also possible to use magnetic stirring rods with a diameter of 3 mm and a length of 6 mm. The stirring power must be increased gradually in order to avoid jamming of the magnetic stirrer.

With the SPECORD 200/210/250 PLUS, two Peltier-tempered cell holders can be used simultaneously for the temperature control of both the sample and the reference. The second cell holder requires a separate control unit for the temperature control. Countercooling is performed by a shared heat exchanger.

Standards

The Peltier-tempered cell holder has been constructed and tested in accordance with the following standards and guidelines:

- DIN EN 61010-1 (IEC 1010-1)
- DIN EN 55011 class B

- DIN EN 61326-1
- DIN EN 61000-3-2
- DIN EN 61000-3-3

Safety instructions

Observe the General Safety Instruction for the operation of the Peltier-tempered accessories (→ "General safety instructions for Peltier-tempered accessories" p. 55).

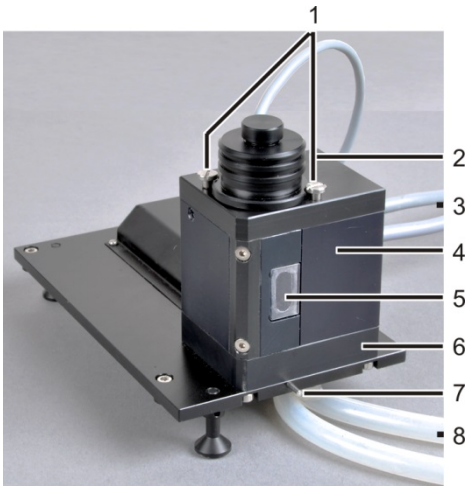
## 17.3.1 Technical data and layout

Technical data

| | |
|---|---|
| Principle | Thermoelectric heating and cooling |
| TEC rear cooling | water-cooled by connection to a heat exchanger |
| Guaranteed controlled temperature range at 25 °C ambient temperature * | -5°C...+ 105°C |
| Preset range for the block temperature | -20°C...+105° |
| Preset accuracy | 0.1 °C |
| Display accuracy | 0.1 °C |
| Control accuracy | +/- 0.1 °C |

* Temperatures below room temperature can cause the cells to mist up if the cover cap is not used. Observe the notes in section "Operation with sample chamber flushing" p. 78.

Layout




1 Locking screws to fasten the cover cap
2 Cover cap
3 Connections to temperature control unit and heat exchanger
4 Cell block with insulation
5 Quartz window
6 Base plate
7 Contact pin
8 Hoses for connection to the heat exchanger

**Fig. 55 Peltier-tempered cell holder with heat exchanger**

## 17.3.2 Installing the cell holder in the sample chamber

### Attention

Lay the connection cables carefully!

The connection cables and water hoses must be laid without tension. Tensile stress on the electrical cables and kinking of the water hoses must be precluded. The water hoses and connection cables must not protrude into the beam path.

Inserting the cell holder in the sample chamber

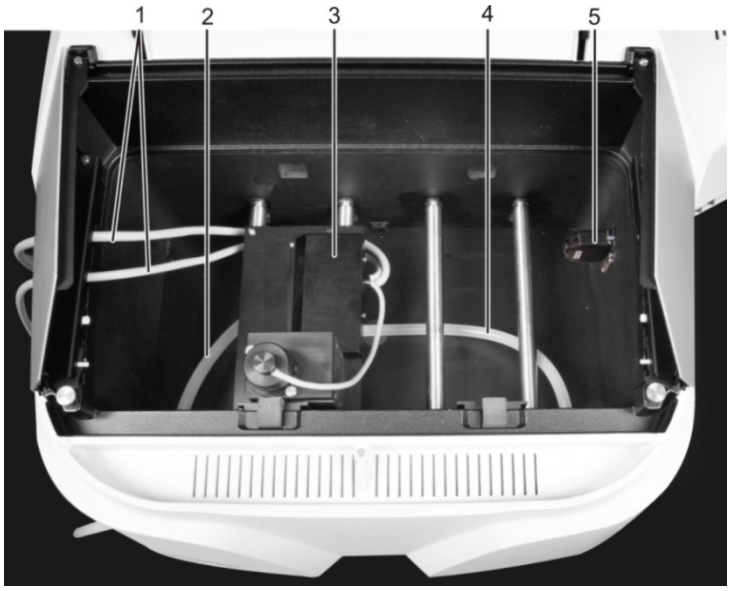



**Fig. 56 Peltier-tempered cell holder installed in the SPECORD PLUS**

1 Cables to the heat exchanger and control unit
2, 4 Hoses for connection to the heat exchanger
3 Base plate
5 Identification connector

1. Screw the support rods in the sample chamber into the top position (→ "Converting the sample chamber" p. 9).
2. Connect the plug to connector (**M**) in the right sample chamber wall.
3. Remove the two cover caps from the apertures of the bottom front panel of the SPECORD PLUS (1 and 3 in Fig. 4 p. 9).
4. Feed the hoses below the support rods through the apertures to the outside.

   To increase light-proofing the apertures in the sample chamber wall have been provided with steps. Should the hose get stuck at these steps when feeding it through, free it up by rotating it slightly. If it is already visible in the outer aperture, bend it towards the aperture with e.g. a pen.
5. Place the cell holder onto the support rods in the measuring channel and slide it up to the stop against the front sample chamber wall.
6. Press down the cell holder on the right side until it engages on the support rods with a click.
7. Connect the hoses to the heat exchanger.
   - Unscrew the knurled nuts from the connection pieces at the heat exchanger and thread it onto the hoses.
   - Slide the hoses onto the connection pieces.
   - Screw the knurled nuts onto the connection pieces and thereby secure the hoses against slipping off.
8. Screw off the left side wall.

9. Remove the two rubbers seals with the larger plugs from the apertures. This is easier if you tilt the rubber seals.
10. Remove the white plugs from the seals and press the electrical cable connections for the heat exchanger and control unit into the slots.
11. Insert the rubber seals into the apertures with the slots pointing down.
12. Screw on the side wall.
13. Connect the connection cables to the control unit and heat exchanger (→"Temperature control unit for Peltier-tempered accessories" p. 82 and "Heat exchanger for Peltier-tempered accessories" p. 86).

Using two cell holders

The SPECORD 200/210/250 PLUS can accommodate two cell holders in the sample chamber. The installation of the second cell holder is largely as described above. For the operation of the second cell holder a second temperature control unit is required.

1. Place both cell holders onto the support rods of the sample chamber.
2. Connect the cell holders to each other using a short piece of hose. Feed the water connection hoses of the cell holders to the outside and connect them to the heat exchanger as described above.
3. Feed the electrical connection cables to the outside as described above. Feed the connections for the cell holder in the reference beam path to the outside through the left sample chamber side.
4. Connect each cell holder to a control unit.
5. Connect the cell holders with the Y adapter supplied to the heat exchanger.


**Fig. 57 Two cell holders (Peltier-tempered with heat exchanger) installed in the sample chamber**

1, 6 Cables to the heat exchanger and control unit
2, 7 Hoses for connection to the heat exchanger
3 Cell holder in the measuring channel
4 Hose connection between the cell holders
5 Cell holder in the reference beam path

Inserting the cell

1. Carefully place the cell into the cell block.
2. Place the cover cap onto the cell so that the grooves of the cover cap fit around the locking screws.

3. Lock the cover cap by rotating it slightly.

   ✓ The cell block is now closed.

Using the cell sensor

The temperature sensing is optionally carried out via the sensor in the cell block or the cell sensor (→ "Using the cell sensor" p. 60).

Flushing the sample chamber

To avoid formation of condensate on the cell walls during operation without cover cap, the sample chamber can be flushed with a dry gas (→ "Operation with sample chamber flushing" p. 65)

### 17.3.3 Temperature control

The Peltier-tempered cell holder is detected during the device initialization with ASpect UV and displayed in the method parameters.

After the temperature control unit is started, the temperature to which the unit is set is established as the start temperature (→ see section "Temperature control unit for Peltier-tempered accessories" p. 82). After starting the measurement all subsequent temperature control is based on the measuring parameters configured in ASpect UV (→ "Temperature control for Peltier-tempered cell holders" p. 61).

### 17.3.4 Care

The Peltier-tempered cell holder is largely maintenance-free.

- Use care when handling the sample substances to avoid contamination, especially inside the cell block.
- Wipe spilled samples or reagents immediately with an absorbent cloth or piece of paper.
- If in spite of all care contamination (sample substances etc.) occurs, the interior of the cell block can be rinsed with ethanol or water with added dishwashing detergent. To drain the rinsing liquid there is an M4 screw with hexagon socket at the bottom of the base plate.
- Store the Peltier-tempered cell holder in the storage container supplied.

## 17.4 Peltier tempered 6- and 8-cell changers

The Peltier-tempered cell changers are automatic sample changing systems. The temperature control of the cell changers is carried out via a separate control unit. The rear of the Peltier elements is kept at a temperature near the ambient temperature by the water cycle of the heat exchanger.

As control sensor a sensor in the top area of the cell block is used. In addition to the control sensor the cell changer features two further sensors for the optional registration of the holder or cell temperature.

The cell sensor is designed specifically for standard cells with round plugs from PTFE and can remain in the cell during the optical measurement.

The cell changers have space for 6 or 8 cells with a pathlength of 10 mm and external dimensions of 12.5 x 12.5 x 45 (L x W x H in mm).

Optionally the cell changers can be equipped with a magnetic stirrer at factory. The magnetic stirrer ensures that an even temperature distribution within the cell is reached more quickly. However, the stirring power is not sufficient to homogeneously distribute e.g. reagents in the sample. Commercially available 10-mm macro cells made of glass, quartz or plastic as well as macro and semi-microcells with cylindrical stirring bottom can be used. Owing to the power limits of the stirrer, only cells with a flat bottom and a bottom thickness of no more than 1.5 mm are suited. The stirring speed is set at the temperature control unit. Optimum stirring of the sample is achieved when using magnetic stirring rods with a diameter of 2 mm and a length of 5 mm. For cells with cylindrical stirring bottom, it is also possible to use magnetic stirring rods with a diameter of 3 mm and a length of 6 mm. The stirring power must be increased gradually in order to avoid jamming of the magnetic stirrer.

Note

The 6- and 8-cell changers differ by the cell block used and thus by the number of cells that can be inserted. However, their operation and installation are identical. Therefore, the two cell changers are described jointly below. For the illustrations the 8-cell changer has been used. The 6-cell changer is installed in the sample chamber in a similar way.

Standards

The Peltier-tempered cell holder has been constructed and tested in accordance with the following standards and guidelines:

- DIN EN 61010-1 (IEC 1010-1)
- DIN EN 55011 class B
- DIN EN 61326-1
- DIN EN 61000-3-2
- DIN EN 61000-3-3

Safety instructions

Observe the General Safety Instruction for the operation of the Peltier-tempered accessories (→ "General safety instructions for Peltier-tempered accessories" p. 55).

## 17.4.1 Technical data and layout

8-cell changer

| | |
|---|---|
| Principle | Thermoelectric heating and cooling |
| TEC rear cooling | air-cooled |
| Guaranteed controlled temperature range at 25 °C ambient temperature * | -5 °C to + 105 °C |
| Preset range for the block temperature | -20 °C to +105 °C |
| Preset accuracy | 0.1 °C |
| Display accuracy | 0.1 °C |
| Control accuracy | +/- 0.1 °C |
| Number of cell positions | 8 |
| Stirrer | Optional |

6-cell changer

| | |
|---|---|
| Principle | Thermoelectric heating and cooling |
| TEC rear cooling | air-cooled |
| Guaranteed controlled temperature range at 25 °C ambient temperature * | -5 °C to 105 °C |
| Preset range for the block temperature | -20°C to +105 °C |
| Preset accuracy | 0.1 °C |
| Display accuracy | 0.1 °C |
| Control accuracy | +/- 0.1 °C |
| Number of cell positions | 6 |
| Stirrer | Optional |

* Temperatures below room temperature can cause the cells to mist up. Observe the notes in section "Operation with cell block flushing" p. 78.

Layout of the cell changer

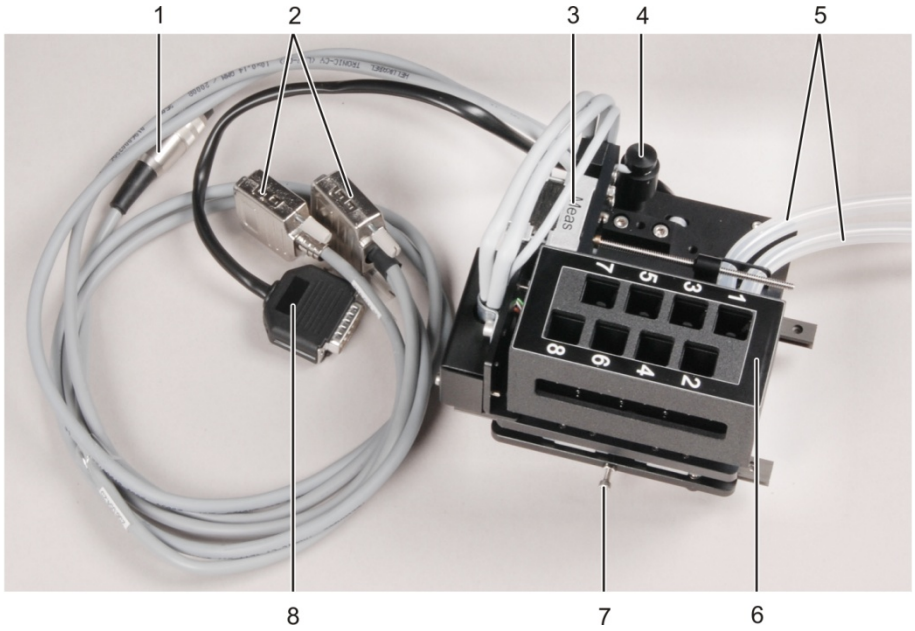



**Fig. 58 Layout of the Peltier-tempered cell changer**

1 Connection to the heat exchanger
2 Connections to the temperature control unit
3 Drive unit with step motor
4 Connection socket for cell sensor in storage position
5 Water hoses
6 Cell block with insulation (with 6 or 8 cell positions)
7 Stop bolt
8 Connection to the SPECORD PLUS

## 17.4.2 Installing, removing and adjusting Peltier-tempered cell changers

### Attention

Lay the connection cables carefully!

The connection cables and water hoses must be laid without tension. Tensile stress on the electrical cables and kinking of the water hoses must be precluded. The water hoses and connection cables must not protrude into the beam path.

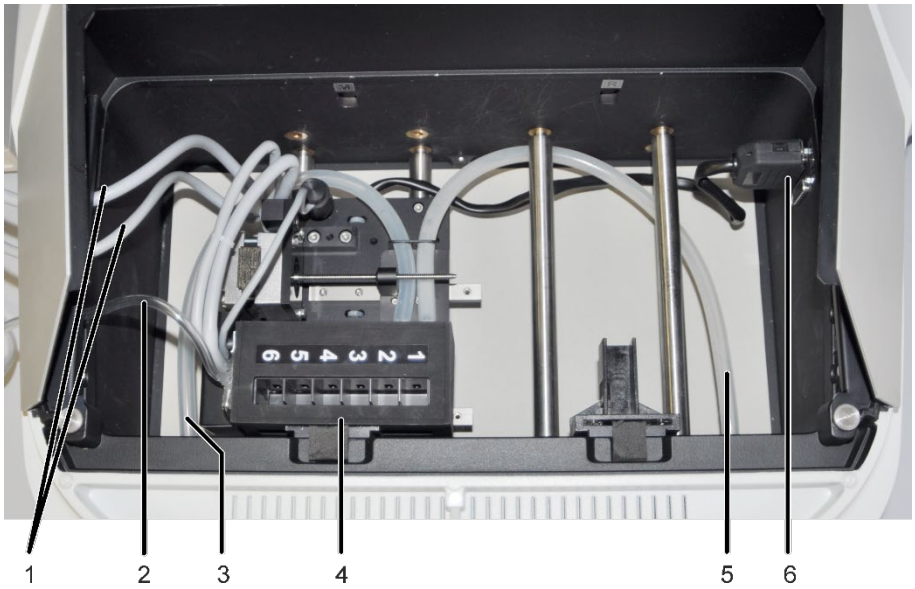



**Fig. 59 Peltier-tempered cell changer installed in the SPECORD PLUS**

1 Electrical cables to the heat exchanger and control unit
2 Hose for the flushing gas
3,5 Hoses for connection to the heat exchanger
4 Cell block with insulation
6 Connector to the SPECORD PLUS

1. Screw the support rods in the sample chamber into the top position (→ "Converting the sample chamber" p. 9).
2. Remove the two cover caps from the apertures of the bottom front panel of the SPECORD PLUS (1 and 3 in Fig. 4 p. 9).
3. Feed the hoses below the support rods through the apertures to the outside.

   To increase light-proofing the apertures in the sample chamber wall have been provided with steps. Should the hose get stuck at these steps when feeding it through, free it up by rotating it slightly. If it is already visible in the outer aperture, bend it towards the aperture with e.g. a pen.
4. Place the cell changer onto the support rods in the measuring channel and slide it up to the stop against the front sample chamber wall.
5. Press down the cell changer on the right side until it engages on the support rods with a click.
6. Connect the hoses to the heat exchanger.
   - Unscrew the knurled nuts from the connection pieces at the heat exchanger and thread it onto the hoses.
   - Slide the hoses onto the connection pieces.
   - Screw the knurled nuts onto the connection pieces and thereby secure the hoses against slipping off.
7. Screw off the left side wall.
8. Remove the two rubbers seals with the larger plugs from the apertures. This is easier if you tilt the rubber seals.
9. Remove the white plugs from the seals and press the electrical cable connections for the heat exchanger and control unit into the slots.

10. Insert the rubber seals into the apertures with the slots pointing down.
11. Lay the hose for the flushing gas in a further rubber seal with matching bore.
12. Screw on the side wall.
13. Connect the connection cables to the control unit and heat exchanger (→"Temperature control unit for Peltier-tempered accessories" p. 82 and "Heat exchanger for Peltier-tempered accessories" p. 86).
14. Plug the connector into the connection (**M**) in the right sample chamber wall.

Adjust cell changers

To position the cells optimally within the beam path the cell changer is adjusted with the aid of a computer.

An adjustment is necessary in case of

- the first use of the cell changer
- after a basic correction
- after transporting the SPECORD PLUS

**Note:** Before adjusting, allow the device to warm up for 2 hours with the lamps switched on.

1. Install the empty cell changer in the sample chamber.
2. Switch the SPECORD PLUS on, start the ASpect UV software and wait until the automatic device initialization is completed.
3. Wait for the warm-up time of 2 hours.
4. Measuring using standard cells or semi-microcells: Perform adjustment with empty cell changer.
   Measurements using microcells for adjusting, place a water-filled microcell in the each of the 6 positions.
5. Start the automatic adjustment by choosing INSTRUMENT ▸ ACCESSORY ▸ ADJUSTMENT.

Using the cell sensor

The software can optionally display either the temperature measured by the sensor in the cell block or by the cell sensor.

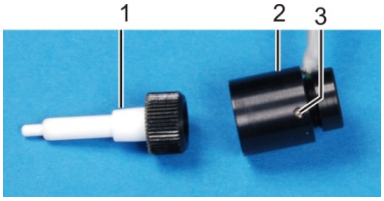
## Attention

**Fragile sensor**

Do not use force when attaching the sensor. It is not necessary to press the sensor in place because it receives enough pressure from the contact pins in the cover cap.

**Flashing temperature display**

If the temperature display (Fig. 65 p. 84) in the control unit is flashing, either the cell sensor is not properly connected to the connection socket or the sensor is faulty.




1 Cell sensor
2 Cover cap of the cell holder
3 Set screw

**Fig. 60 Cell sensor for Peltier-tempered cell changers**

1. Seal a standard cell with round plug with the cell sensor supplied.
2. Insert the cell into the cell block and close it with the cover cap.
3. Turn the change-over switch at the control unit to "cell".

Operation with cell block flushing

Depending on the room temperature and the relative humidity, the water of the ambient air condensates below a certain block or cell temperature (dew point) on the cell walls and the cell block. This distorts the measuring results.

The following context applies for the start of condensate formation (dew point temperature):

$$\vartheta_K = \left(\frac{\text{relativeLuftfeuchte}}{100}\right)^{0,1247} * (109,8 + \vartheta_R) - 109,8$$

$\vartheta_K$ - dew point temperature in °C

$\vartheta_R$ - room temperature in °C

For a room temperature of 20 °C and a relative humidity of 60 % the dew point temperature is 12 °C. The condensate formation can be avoided by flushing the sample chamber with dry gas (air, nitrogen or argon) via the flushing gas hose. The following gas flow rates must be assured:

| | |
|---|---|
| 6-cell changer: | 400 l/h |
| 8-cell changer: | 200 – 300 l/h |

At these gas flow rates, even slight condensate films are removed from cells that were pre-cooled when they were inserted.

**Note:** Before working at low temperatures, the sample chamber should be pre-dried. Clean out the sample chamber by operating the Peltier cell changer at +80 °C for approx. 10 minutes.

Removing the cell changer from the sample chamber

1. Switch off the heat exchanger and the temperature control unit and disconnect them from the mains supply.
2. To move the cell changer to the parking position: Choose INSTRUMENT ▸ ACCESSORY ▸ SAMPLE POSITION / button: [PARKING].
3. To switch off the accessory: Choose INSTRUMENT ▸ ACCESSORY ▸ ACCESSORY OFF.
4. Disconnect the plug from the connector in the sample chamber wall.
5. Disconnect the hoses from the heat exchanger.
6. Allow the water to drain from the hoses and collect the water.
7. Pull the hoses from the apertures in the front sample chamber wall.
8. Place the cover caps onto the apertures in the front panel of the SPECORD PLUS (1 & 3 in Fig. 4 p. 9) to restore the light-proofing.
9. Unscrew the side component of the sample chamber.

10. Remove the rubber seal from the aperture in the left sample chamber wall and pull the lines out of the slot in the rubber seal.
11. Close the opening in the rubber seal with the white plug.
12. Insert the rubber seal into the sample chamber aperture with the slot pointing down.
13. Screw on the side component.
14. Pull up the cell changer on the right side of the base plate and lift it out of the sample chamber.
15. Package the cell changer in the storage container.


**Fig. 61 Peltier-tempered 8-cell changer in the storage container**

### 17.4.3 Temperature control

The Peltier-tempered cell holders are detected during the device initialization with ASpect UV and displayed in the method parameters.

After the temperature control unit is started, the temperature to which the unit is set is established as the start temperature (→ see section "Temperature control unit for Peltier-tempered accessories" p. 82). After starting the measurement all subsequent temperature control is based on the measuring parameters configured in ASpect UV (→ "Temperature control for Peltier-tempered cell holders" p. 61).

### 17.4.4 Using two Peltier-tempered cell changers

With the SPECORD 200/210/250 PLUS, two Peltier-tempered 6- or 8-cell changers can be used simultaneously to increase the possible number of samples or to have a reference for each sample. The second cell changer requires a separate temperature control unit. Countercooling is performed by a shared heat exchanger.

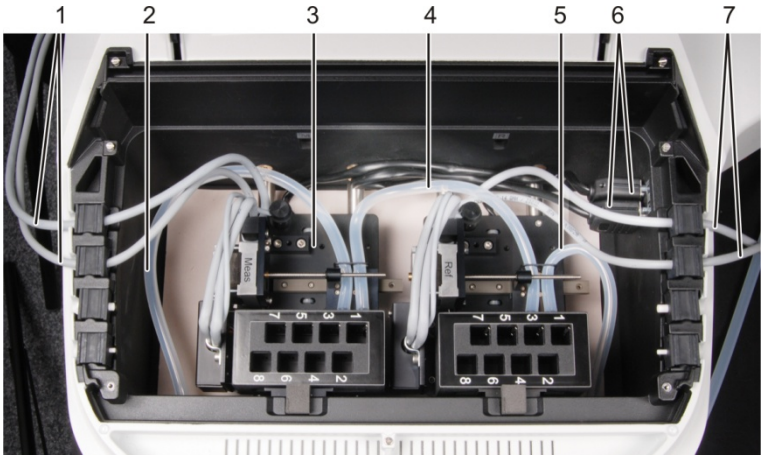



**Fig. 62 Two Peltier-tempered 8-cell changers installed in the sample chamber**

1 Connection cables of the cell changer in the measuring channel
2 Water hose
3 Cell changer in the measuring channel
4 Connection hose between two cell changers
5 Water hose
6 Connector
7 Connection cables of the cell changer in the reference beam path

Installing two cell changers

The installation of the second cell changer is largely as described in section "Installing" p. 75.

1. Connect the cell changers to each other using a short piece of hose.
2. Feed the water connection hoses of the cell changers to the outside and connect them to the heat exchanger.
3. Place both cell changers onto the support rods in the sample chamber and slide them to the front sample chamber wall.
4. Using the guide rail (5 in Fig. 34) (included), align both cell changers with respect to each other so that they have the same distance from the front sample chamber. Correct the adjustment of the stop bolts (7 in Fig. 58 p. 75), if required.
5. Press down the base plates of the cell changers until they engage on the support rods.
6. Connect the two cell changers to the connections in the right sample chamber wall: the cell changer in the measuring channel to (**M**) and the cell changer in the reference channel to (**R**).
7. Lay the electrical connection cables and the hoses for the flushing gas as described above. Feed the connections for the cell changer in the reference channel to the outside through the right sample chamber side.
8. Connect each cell changer to a control unit.
9. Connect the cell changers with the Y adapter supplied to the heat exchanger.
10. Adjust the cell changers (→ "Installing, removing and adjusting Peltier-tempered cell changers" p. 75).
11. Attach the "M" and "R" labels to the step motors of the cell changers in the measuring and reference beam paths, making sure they clearly visible.

    **Note:** If the cell changers are alway set to the same position, the adjustment does not need to be repeated.

Measuring with two cell changers

The configuration for the use of the cell changers for synchronous and offset mode can be found in chapter "Carrying out measurements with two cell changers" p. 53. The temperature configuration is explained in section "Temperature control for Peltier-tempered cell holders" p. 61.

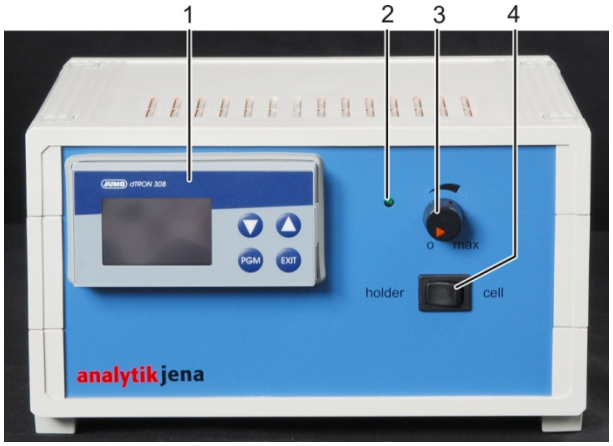
## 17.5 Temperature control unit for Peltier-tempered accessories

The control unit implements the temperature control for the Peltier-tempered accessories.

Technical data

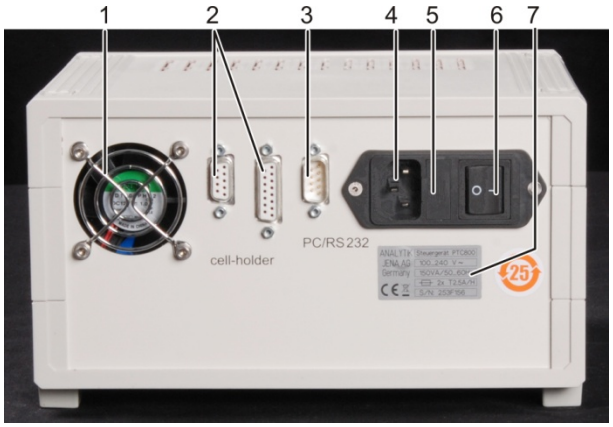
| | |
|---|---|
| Control unit mass | 2.5 kg |
| Dimensions (W x H x D) | 225 x 130 x 200 mm³ |
| Mains voltage | 100 – 240 V |
| frequency | 50 ... 60 Hz |
| Power consumption | 75 VA |
| Mains fuses | 2 x T 2.5 AH / 250V, type 19181 by Wickmann |
| EMC (interference emission and interference resistance) according to DIN EN 61326 and 61326/A1 | The device may be positioned and operated anywhere. |
| Fire resistance of the control unit housing according to UL94 | HB / 1.6 |
| Protection type | IP 20 |
| Data connection | RS232 interface |
| Operating temperature | +15°C...+35°C |
| Transport and storage temperature | -40°C...+60°C |
| relative humidity | up to 90% (at +30°C) |

Layout




1 Controller insert with display and keys
2 Status light for heat heat exchanger
3 Controller for the speed adjustment of the magnetic stirrer (only for accessories with stirrer)
4 Change-over switch for holder or cell temperature

**Fig. 63 Displays and switches at the temperature control unit**




1 Fan
2 Connections of the cell changers / holders
3 Connection to the SPECORD PLUS
4 Mains connection
5 Holder for mains fuses
6 Mains switch
7 Type plate

**Fig. 64 Connections on the rear of the temperature control unit**

Connecting the temperature control unit

1. Connect the 9- and 15-pin connector of the cell holder / changer to the corresponding connections at the rear of the control unit.
2. Connect the RS232 interface of the control unit with the ACC1 interface of the SPECORD PLUS.

   If two cell holder / changers are used, connect the temperature control units as follows:

   - Temperature control unit for the measuring channel: Interface ACC1
   - Temperature control unit for the reference channel: Interface ACC2
3. Connect one mains cable to the power inlet and then to the mains socket.
4. Switch on the temperature control unit by the mains switch on the rear.

Using a temperature control unit with heat exchanger

Not for an air-cooled cell holder

The LED at the front of the control unit (2 in Fig. 63) indicates the three operating states of the heat exchanger:

| | |
|---|---|
| LED off | No heat exchanger present or heat exchanger not connected. |
| LED red | The heat exchanger is connected but not switched on. |
| LED green | The heat exchanger is connected and switched on. |

If the Peltier-tempered accessory is operated without heat exchanger or with the heat exchanger switched off, cooling is electronically suppressed, but heating is possible. At constant ambient temperature and with a setpoint setting of approx. 5 °C above the sample chamber temperature the specified target values can with increased control cycles be maintained with an accuracy of 0.1 – 0.2 °C. However, the control accuracies specified in the sections "Technical Data" relate to operation with heat exchanger.

Setting the temperature control unit

After the temperature control unit is started, the temperature to which the unit is set is established as the start temperature. After starting the measurement all subsequent temperature control performed by the ASpect UV software (→ "Temperature control for Peltier-tempered cell holders" p. 61).

The start temperature can be changed at the temperature control unit. However, this setting is not required for measurements.


1 Block temperature display
2 Arrow keys to change the temperature setting
3 EXIT key
4 PGM key
5 Temperature display for holder or cell

The numbers 1 – 5 are status indications of the controller and only relevant for service.

**Fig. 65 Display and keys for temperature adjustment at the control unit**

## Attention

The specified set temperature relates to the block temperature. Depending on the sample chamber temperature, the type of cells used and the type and quantity of sample material, the resulting final values of the inside cell temperature and time periods until they are reached will be different (Fig. 66).

1. Press the PGM key until "SP 1" appears in the display for the holder or cell temperature. Press it again until "SP 1" flashes.
2. Use the arrow keys to specify the target temperature.
3. Confirm the target temperature by pressing the PGM key. After a few seconds the set value will automatically be accepted.
4. Exit the entry level by pressing the EXIT key twice.

Alternatively, the temperature setting can be made directly via the arrow keys:

1. Press one of the two arrow keys until the setting for the block temperature changes.
2. Use the arrow keys to specify the target temperature.
3. Wait a few seconds until the values is automatically accepted.

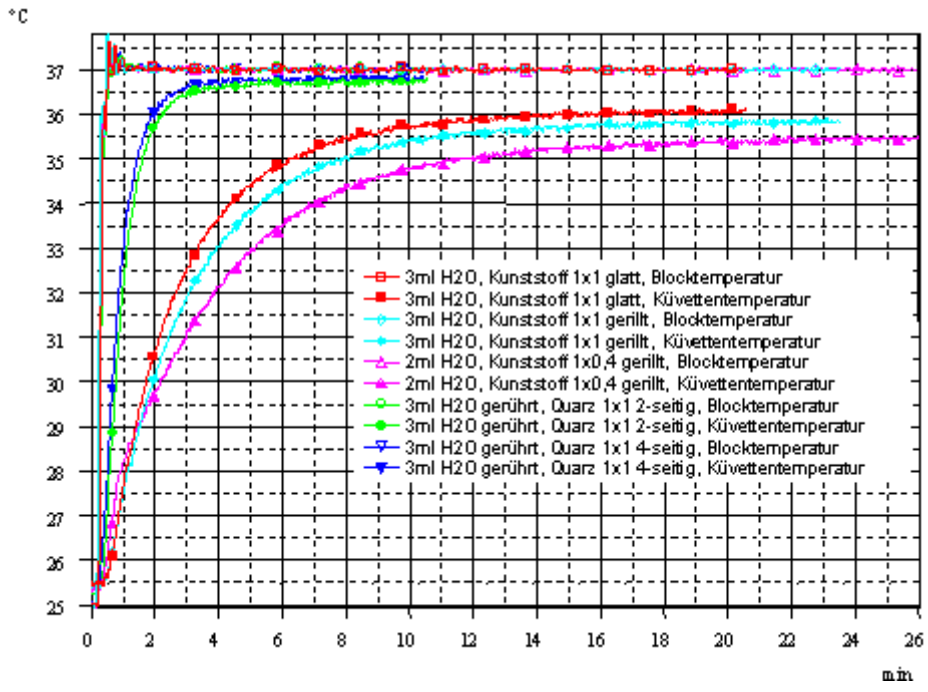


**Fig. 66 Temperature curve in the block and inside the cell for different cells**

Registering the cell temperature

When the target is reached at the control unit, the cell temperature is determined by the cell sensor and assigned to the measurement value (A/%T).

1. Use cells with an opening for round plugs only. Close the cell with the cell sensor supplied. Slide the connection socket onto the cell sensor.
2. Turn the change-over switch at the control unit to "cell".
3. If the cell sensor is no longer required pull the cell sensor out of the connection socket. Turn the change-over switch at the control unit back to "holder".

## Attention

If the temperature display in the control unit flashes, either the cell sensor is not properly connected to the connection socket or the sensor is faulty.

Replacing fuses

You can replace defective fuses as follows:

1. Disconnect the mains cable from the control unit connection.
2. Pull out the fuse holder (5 in Fig. 64) by the lid.
3. Replace the defective fuses. Use the following fuses:

   2 x T 2.5 AH / 250V, type 19181 by Wickmann
4. Close the fuse holder.
5. Connect the mains cable to the control unit connection.

## 17.6 Heat exchanger for Peltier-tempered accessories

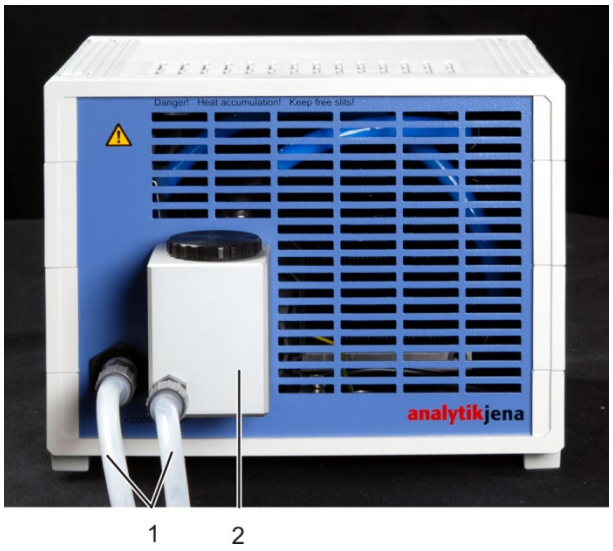
### Attention

Avoid overheating Always keep the air vents unobstructed.

Technical data

| | |
|---|---|
| Mass without refrigerant | 3.2 kg |
| Dimensions (W x H x D) | 225 x 175 x 200 $mm^3$ |
| Mains voltage | 220 - 240 V (-15 % / +10 %) |
| frequency | 50 Hz |
| Power consumption | 50 VA |
| Mains fuses for WC 601 | 2 x T 1.6 AH / 250V, type 19181 by Wickmann |
| EMC (interference emission and interference resistance) according to DIN EN 61326 and 61326/A1 | The device may be positioned and operated anywhere. |
| Fire resistance of the heat exchanger housing according to UL94 | HB / 1.6 |
| Protection type | IP 20 |
| Coolant | approx. 0.4 l distilled water with 4 ml isopropanol added |
| maximum delivery height | 1.2 m |
| Operating temperature | +15°C to +35°C |
| Transport and storage temperature | -40°C to +60°C |
| relative humidity | up to 90 % (at +30 °C) |

Layout




1 Water hoses of the Peltier-tempered accessories
2 Compensation container with cover

**Fig. 67 Heat exchanger – hose connections and compensation container**


1 Type plate
2 Connection to the Peltier-tempered accessories
3 Mains switch
4 Fuse holder
5 Mains connection

**Fig. 68 Heat exchanger – connections on the rear**

Preparing the refrigeration fluid

The following is needed to produce the coolant:

- Distilled water or de-ionized water
- Isopropanol
- A vessel for mixing the coolant

Distilled water: Mix approx. 400 ml distilled water with 4-5 ml isopropanol.

De-ionized water: Mix 350 ml deionized water with 50 ml isopropanol.

Connecting the heat exchanger

1. Install the cell holder/changer in the sample chamber and connect the water hoses to the heat exchanger.
2. Connect the connector of the cell holder/changer to the corresponding connection at the rear of the control unit.
3. Connect one mains cable to the power inlet and then to the mains socket.
4. Unscrew the cover (anti-clockwise).
5. Switch on the heat exchanger by the mains switch.
6. Through the opening in the compensation container fill the prepared coolant until a filling level of approx. 1.5 cm below the top edge of the compensation container has been reached with bubble-free circulation (no air bubbles rising in the tank and noise-free circulation of the coolant).
7. Close the compensation container finger-tight with the cover.

## Attention

The maximum delivery height of the heat exchanger is approx. 1.2 m.

Dependent on the location of the heat exchanger, the routing of the hoses and for design reasons air cushions may form during the system filling process and only dissipate after a few minutes. Brief switching off and on can accelerate the dissipation of this air cushion.

### Replacing the fuses at the heat exchanger

You can replace defective fuses as follows:

1. Disconnect the mains cable from the heat exchanger connection.
2. Pull out the fuse holder (4 in Fig. 68) by the lid.
3. Replace the defective fuses. Use the following fuses:

   2 x T 2.5 AH / 250V, type 19181 by Wickmann
4. Close the fuse holder.
5. Connect the mains cable to the heat exchanger connection.

# 18 Cassette sipper system

The cassette sipper system rationalizes the manual laboratory work for medium sample series. The sample supply is computer-controlled. Using a hose pump the sample is transported into the flow cell for measuring and pumped after the measurement into the waste bottle.

The sample supply can be manually or using an autosampler.

The cassette sipper system can also aspire the sample and reference simultaneously. In this case equip the pump with two hose cassettes and place the reference cell into an adjustable cell holder in the reference beam path of the SPECORD PLUS.

The cassette sipper system is suitable for cells with the following dimensions:

| | |
|---|---|
| Pathlength | 10, 20, 40 and 50 mm |
| Cell width | 12.5 mm |
| Radiation height | 8.5 – 15 mm |

The cassette sipper system must be installed in the sample chamber and connected before the device initialization of the SPECORD PLUS takes place. During the device initialization it is automatically detected and its specific configurations are unlocked in the method parameters.

Layout

The cassette sipper system consists of the following components:

- adjustable cell holder for 10, 20, 40 and 50 mm pathlength
- integrated ISMATEC pump head with two hose cassettes
- hoses for sample supply, for the pump and for the sample discharge into the waste bottle
- flow cell (to be ordered separately

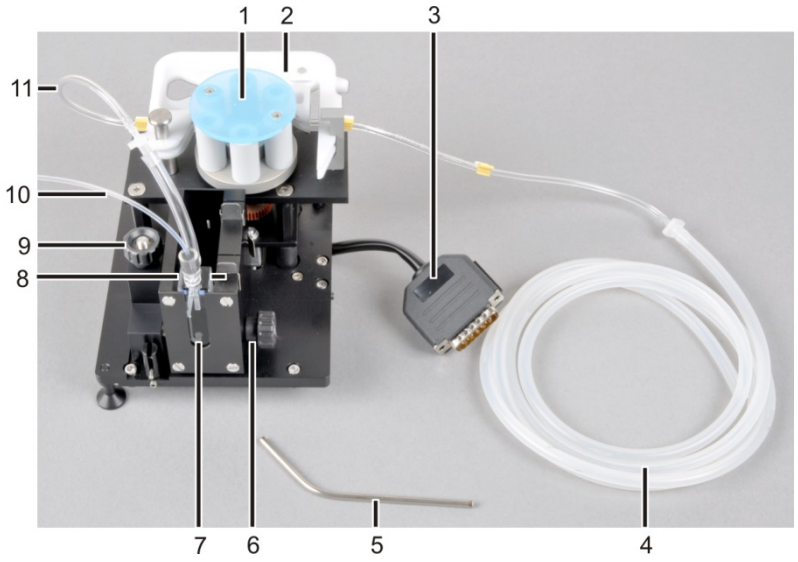
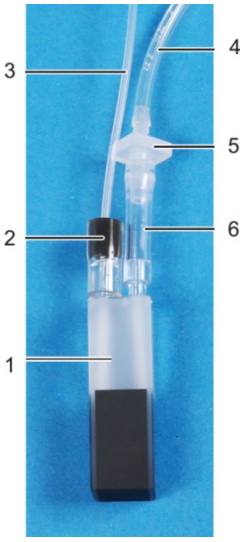



**Fig. 69 Layout of the cassette sipper system**

1 Pump head with pump rolls
2 Hose cassette with eccentric lever
3 Connector
4 Drainage hose
5 Cannula
6 Horizontal adjustment
7 Flow cell
8 Slider with leaf spring
9 Vertical adjustment
10 Sample hose
11 Pump hose

## 18.1 Installing the cassette sipper system

1. Screw the support rods into the top position (→ "Converting the sample chamber" p. 9).
2. Attach the sample hose to the intake connection of the flow cell. The flow direction is indicated on the cell by an arrow.
   Slide the Tygon hose piece over the intake connection. The black Viton seal must sit on the intake connection and Teflon hose must reach into the cell. This prevents accumulation of sample at the intake connection.
3. Attach the pump hose to the drain connection of the cell.
   - Cut a piece of approx. 3 cm length off the drain hose.
   - Slide the hose piece onto the drain connection.
   - Connect the hose piece to the pump hose using a hose coupling.




1 Flow cell
2 Tygon hose with Viton seal
3 PTFE sample hose
4 Pump hose
5 Hose coupling
6 Piece of drain hose

**Fig. 70 Flow cell with connected sample hose and pump hose**

4. Connect the pump hose at the other end with the drain hose using a hose coupling.
5. Clamp the pump hose between the first and second stopper into the hose cassette.

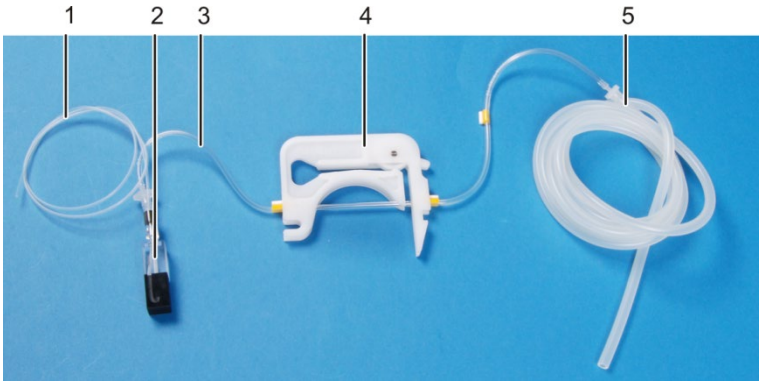



**Fig. 71 Complete hose kit for the cassette sipper system**

1 Sample hose
2 Flow cell
3 Pump hose
4 Hose cassette
5 Drainage hose

6. Insert the slider with the leaf spring (8 in Fig. 69) at the location corresponding to the cell pathlength.

   **Note:** For cells with a pathlength of 40 or 50 mm use both sliders in order to align the long cell in parallel with the beam path.
7. Insert the cell into the holder. The drain connection points towards the pump.
8. Attach the hose cassette to the pump head (Fig. 69).
9. Using the eccentric lever press the pump hose against the rollers until it is squeezed completely. (2 in Fig. 69 p. 90)
10. Unscrew the left side wall of the sample chamber.
11. From the outside, feed the cannula through the smallest aperture without removing the rubber seal.

12. Remove the cover caps from the left and right sample chamber aperture in the front panel of the SPECORD PLUS (1 & 3 in Fig. 4 p. 9).

13. Lay the drain hose under the support rods through the aperture to the outside and hang the end into a suitable waste container.

    To increase light-proofing the apertures in the sample chamber wall have been provided with steps. Should the hose get stuck at these steps when feeding it through, free it up by rotating it slightly. If it is already visible in the outer aperture, bend it towards the aperture with e.g. a pen.

14. Place the cassette sipper system onto the support rods in the measuring channel. The cell holder must point forward towards the receiver.

    Press down the cassette sipper system on the right side until it engages on the support rods with a click.

15. Connect the connector to connection (**M**) in the right sample chamber wall.

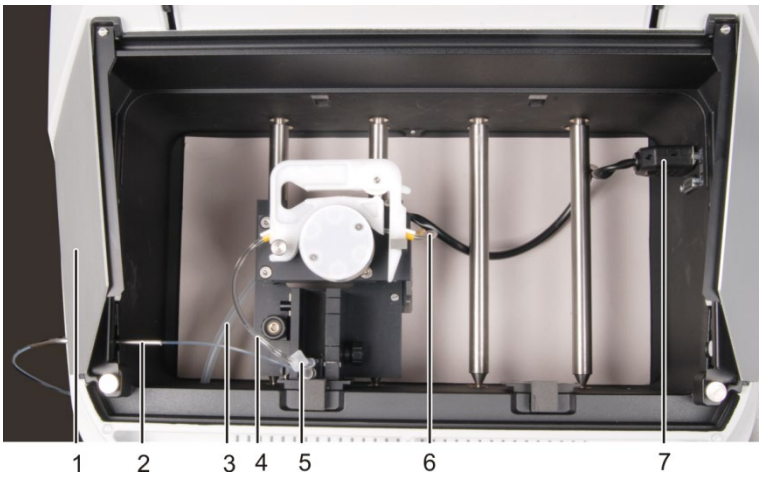



**Fig. 72 Cassette sipper system installed in the sample chamber**

1 left sample chamber wall
2 sample cannula
3 Drainage hose
4 pump hose, connected to cell
5 Flow cell
6 pump hose, connected to drainage hose

Using the aperture baffle

The cassette sipper system is fitted with an aperture baffle. The baffle is located on the rear of the cell duct and is attached by means of four screws. It prevents distortion of the measuring result by light that passes through the cell walls or through air bubbles on the cell walls. The baffle is particularly suitable for working with cells that have a large pathlength as well as cells with a small aperture and non-blackened rims. The aperture baffle is adjusted for a radiation height of 15 mm but can be adapted for a radiation height of 8.5 mm.

If cells with large apertures are used, the aperture baffle is removed in order to achieve a higher light level and thus a better signal-to-noise ratio.

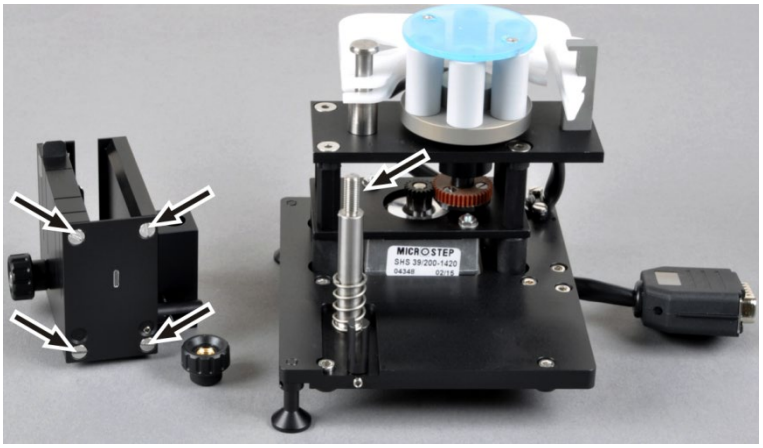



**Fig. 73 Aperture baffle on the rear of the cell duct**

1. Unscrew the vertical adjustment screw and remove the cell duct from the cassette sipper system.
2. To remove the aperture baffle: Unscrew the fours screws at the rear of the cell duct and remove the aperture baffle.
3. To adapt the aperture baffle to a radiation height of 8.5 mm: Fasten the aperture baffle in the lower position on the rear of the cell duct (see Fig. 23 p. 28).
4. Re-attach the cell duct to the cassette sipper system.

## 18.2 Adjusting the flow cell and determining the pumping time

Adjusting the flow cell

To position the cells optimally within the beam path the cell changer can be adjusted. An adjustment is necessary in case of

- the first use of the cassette sipper system
- after a basic correction
- after changing the flow cell.

1. Insert a water-filled flow cell into the cassette sipper system.
2. Initially, adjust visually:
   - For adjustment, configure the zeroth order of the Vis lamp (→"Configuring the zeroth order" p. 11).
   - Insert a paper strip of approx. 10 mm width into the opening for cloudy samples and observe the traced light beam from above.
   - Consecutively turn the screws for vertical and horizontal adjustment (9 and 12 in Fig. 69) until the light hits the paper strip at the center.
   - Remove the paper strip.

3. In the PHOTOMETRY module set a method with the following parameters:

   GENERAL tab:

| Parameter | |
|---|---|
| MEAS. MODE | TRANSMITTANCE |
| WAVELENGTHS [NM] | 500.00 |
| INTEGRATION TIME [S] | 0.1 |
| SLIT [nm]<br>(SPECORD 210/250 PLUS only) | 1 |

   SAMPLE SEQUENCE tab:

   - One reference measurement in first position
   - Further sample rows with the sample type SAMPLE for adjustment

4. Start the measurement with  .
5. Perform the reference measurement.
6. Consecutively change the vertical and horizontal adjustment and each time measure the transmission **with the sample chamber cover closed**. Repeat the process until the transmission value reaches its maximum.

   **Note:** Do not modify the vertical adjustment and the horizontal adjustment at the same time.

### Determining the optimum pumping time for the cassette sipper system

The determination of the optimum pumping time is a process whereby measured values (energy, absorbance and transmission) are recorded while sample or reference solution is pumped through the cell. The change of the measured value over time is displayed on the screen. During the process the measured value increases or decreases and finally reaches a plateau. The optimum pumping time corresponds to the reaching of the plateau phase. At this time the cell has been flushed sufficiently and the carry-over is smallest.

1. Provide reference and sample solutions.
2. Start the optimization of the pumping time by choosing INSTRUMENT ▸ ACCESSORY ▸ OPTIMIZE PUMP TIME.
3. Define the parameters for the optimization of the pumping time on the PARAMETERS tab:

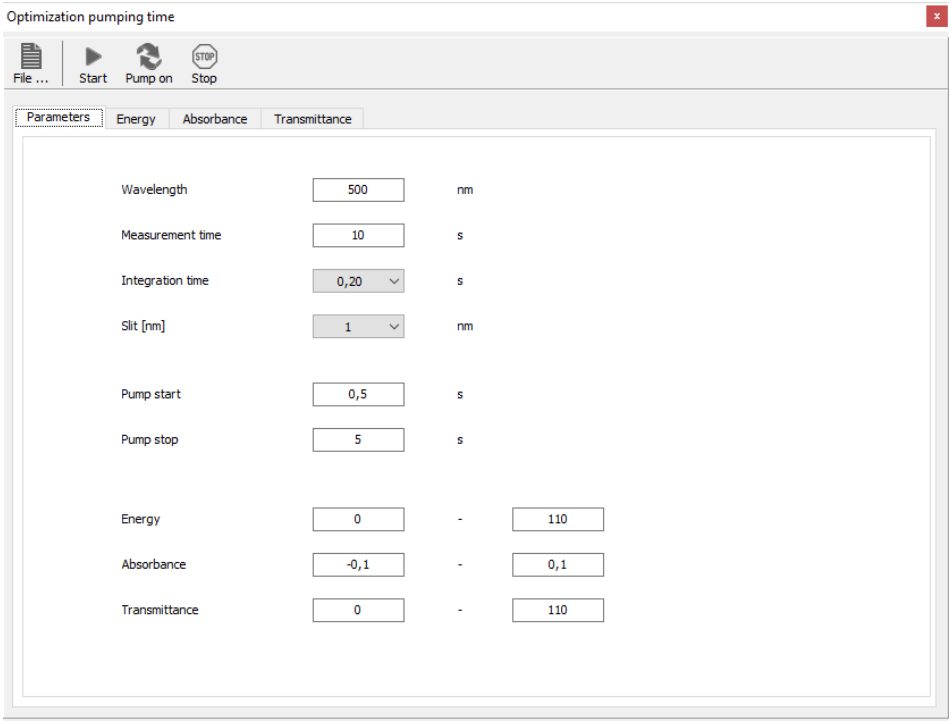



**Fig. 74 Measuring parameter configuration for the pumping time**

| Parameter | Description |
|---|---|
| WAVELENGTH | Analysis wavelength of the sample |
| MEASUREMENT TIME | Total measuring time for the optimization |
| INTEGRATION TIME | Time for recording a measuring point, e.g. 0.1 s. |
| SLIT | only for SPECORD 210/250 PLUS<br>Slit setting, e.g. 1 nm. |
| PUMP START | Time during the measurement at which the pump starts |
| PUMP STOP | Time during the measurement at which the pump stops |
| ENERGY | Ordinate range for the display of the measured values as energy values |
| ABSORBANCE | Ordinate range for the display of the measured values as absorbance values |
| TRANSMITTANCE | Ordinate range for the display of the measured values as transmission values |

4. Remove the flow cells from the holder and fill them with reference solution:
   - Submerge the aspiration hose in the reference solution and start the pump by clicking on .
   - Once the cell is filled without bubbles, stop the pump by clicking on .
5. Place the flow cell back in the holder and close the sample chamber cover.
6. Submerge the aspiration hose in the sample solution and start the measurement by clicking on .

7. Observe the change of the measured value on either one of the tabs ENERGY, ABSORBANCE or TRANSMITTANCE:
   - The time range during which the pump is running is shown on a white background. The range during which the pump was stopped is shaded in gray.
   - The pump time is optimal if the measured value is stable or has reached the required accuracy.
   - During the time after stopping the pump it can be observed whether the measured value remains stable or still changes due to decreasing turbulence in the cell.
8. Take a note of the pumping time determined in this way in order to adopt it for the method at a later stage.
9. Optionally, save the optimization information by clicking on  .

## 18.3 Measuring with the cassette sipper system

Configurations in the measuring parameters

Make the following settings under Method on the ACCESSORY tab:

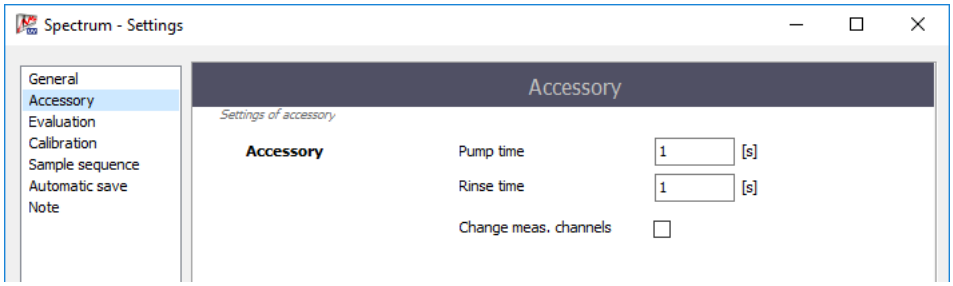



**Fig. 75 Measuring parameter configurations for the cassette sipper system**

| Option | Description |
|---|---|
| PUMPING TIME [S] | Enter the optimum pumping time determined. |
| RINSE TIME [S] | Time during which rinse samples are pumped through the flow cell<br>No measurements are made for rinse samples. The optimum pumping time depends on the characteristics of the sample and of the rinse fluid. |
| CHANGE MEAS. CHANNELS | Swap the function of the beam paths in the sample chamber.<br>Enabled: The cassette sipper system is installed in the R beam path of the sample chamber. |

In the sequence, you can specify rinse samples before or after the sample measurement. To do so, insert a row in the desired position and select the WASH sample type.

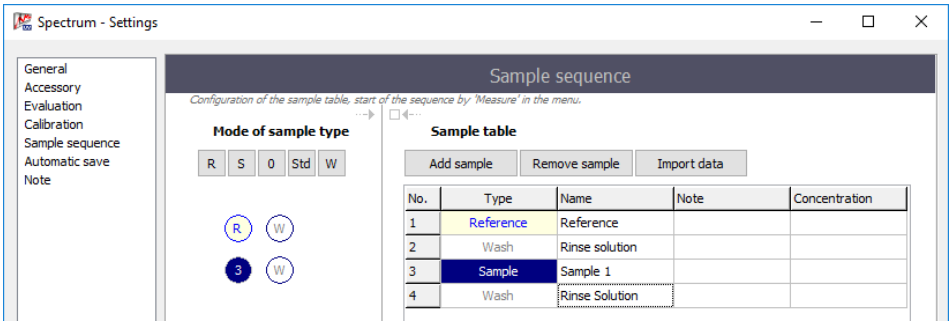



**Fig. 76 Sequence with rinse steps**

Running the measurement

- After starting the measurement, follow the instructions on the screen and move reference solution, sample or rinse sample to the aspiration hose as prompted.
- The / button in the menu bar can be used to switch the pump on and off independently of the measurement in order to rinse, empty or fill the system.
- It is recommended to rinse the system for a few minutes after processing a sequence.

## 18.4 Care and maintenance

The maintenance for the cassette sipper system is limited to replacing the pump hoses. Pump hoses made of different materials or with different inside hose diameters are available from our customer service.

Please observe the following:

- Avoid contamination of the cassette sipper system. Wipe spilled samples or reagents immediately with an absorbent cloth or piece of paper. Remove stubborn contamination with a soft cloth wetted with a commercial dishwashing detergent.
- After completing the work fill the flow cell with distilled water. If the cell dries out completely any sample residue might get stuck in the flow cell.
- After completing the work, detach the hose cassette from the pump in order to relieve the pump hoses. This ensures that the pump hoses remain elastic for longer.
- Before removing the cassette sipper system from the sample chamber, always switch it off via software control (INSTRUMENT ▸ ACCESSORY ▸ ACCESSORY OFF) or switch the SPECORD PLUS off by the mains switch. The accessory can then be removed from the sample chamber without risk of short circuit.

# 19 Autosampler APG

The APG is a xyz sampler. It is used in conjunction with the cassette sipper system for the sample supply to a flow cell in the SPECORD PLUS.

From a flushing container flushing liquid can be taken to clean the flow system.

Sample trays are available for the following numbers and volumes of samples:

| Number of samples | Volume of the sample containers | included in the scope of delivery |
|---|---|---|
| 18 | 100 ml | optional (Model APG S) |
| 49 | 50 ml | optional |
| 64 | 30 ml | Standard |
| 116 | 12 ml | optional |

The APG S sampler can also be used for other sample container types that differ from the standard sample containers with respect to the number and volume. The software settings need to be adjusted accordingly.

### WARNING

Danger of electric shock!

Always disconnect the mains plug before opening the housing. Make sure that liquids do not get into the interior of the device or in contact with cable connections.

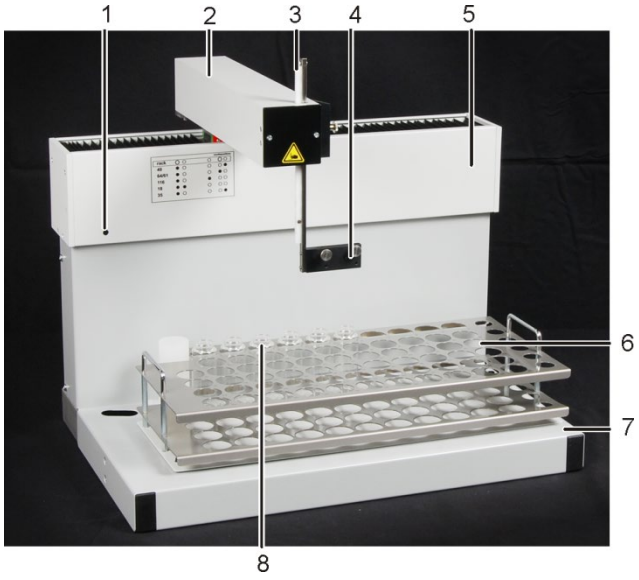
### CAUTION

Risk of crushing!

Keep your hands out of reach of the sampler arm and of the cannula while measuring is in process.

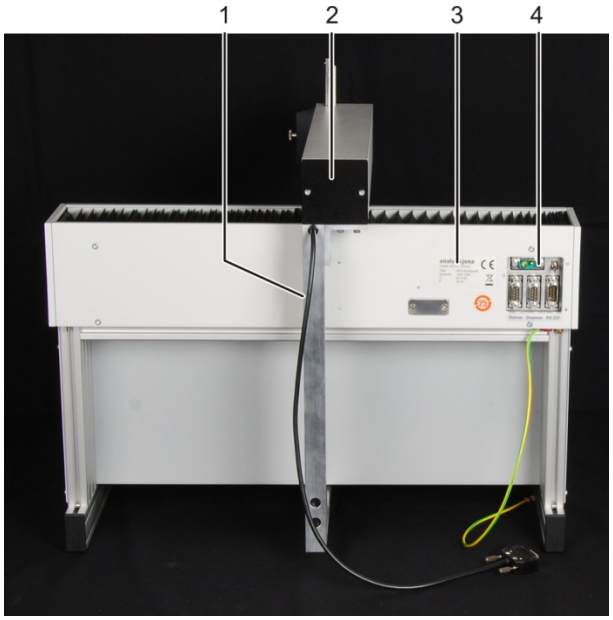
When setting up the device, make sure there is enough space for the movement range of the autosampler arm. The sampler arm also moves towards the rear!

Layout



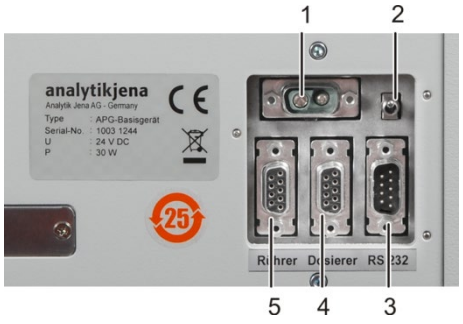

1 Control LED
2 Sampler arm
3 Cannula guide
4 Universal cannula holder
5 Basic device
6 Sample tray with 64 samples
7 Base with floor frame
8 Sample containers

**Fig. 77 Layout of the APG with sample tray for 64 samples**




1 Stirring arm
2 Sampler arm
3 Type plate
4 Electrical connections

**Fig. –78 Layout of the APG - rear panel**



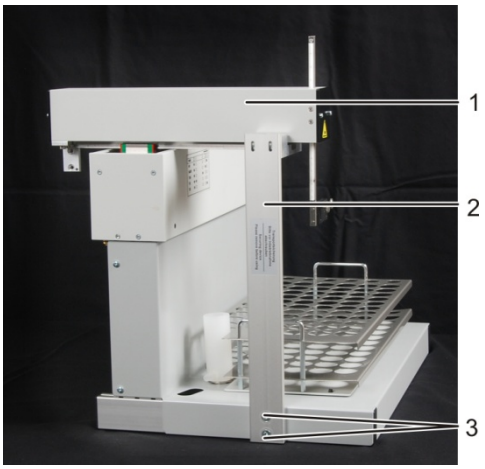

1 Connection to the power supply
2 Device switch
3 Connection to the SPECORD PLUS (ACC2)
4 Not used
5 Connection to stirrer

**Fig. 79 Electrical connections at the APG**

## 19.1 Installing and commissioning the APG

Removing the transport lock

1. Take the autosampler and the accessories out of the packaging and place onto a level laboratory surface.
2. Remove the transport lock:
   - Remove the two countersunk screws with the A/F3 hexagon head wrench supplied.
   - Remove the complete transport retaining clip and retain the transport lock well (for transport in case of a service requirement etc.)




1 Sampler arm
2 Transport retaining clip
3 Screws

**Fig. 80 Transport lock at the APG**

Fitting the stirring arm

**Note:** The stirring function can only be used with the sample trays for 49 and for 64 samples and optionally with the APG S autosampler. If the tray for 116 samples is used, it is not necessary to fit the stirring arm.

Fitting the stirring arm to the angle bracket at the rear end of the sampler arm:

1. Screw on the arm with the countersunk screws supplied (DIN 7991-M4x10) using the A/F2.5 hexagon head wrench.
2. Tighten the screws evenly to allow the arm to be aligned.
3. Connect the stirrer cable to the "Stirrer" port on the rear of the autosampler.


1 Bracket at the sampler arm
2 Countersunk screws
3 Stirring arm

**Fig. 81 Fitting the stirring arm to the APG**

Inserting the cassette sipper system

1. Place the cassette sipper system in the measuring channel of the sample chamber (→ "Installing the cassette sipper system p. 90).

   Plug the connector of the cassette sipper system into connection (**M**) in the right sample chamber wall.

2. Connect the sample intake hose to the flow cell and feed it through one of the apertures in the sample chamber wall to the outside.
3. Switch the SPECORD PLUS on, adjust the flow cell in the beam path (→ "Adjusting the flow cell and determining the pumping time" p. 93), switch the SPECORD PLUS off.

Setting up the APG

1. Connect the low voltage side cable of the table power supply unit to the rear of the autosampler.

   Do not connect the table power supply unit to the mains yet.
2. Place the APG with a minimum distance of 10 cm to the left of the SPECORD PLUS.
3. Connect the identification connector of the APG to the connector (**R**) in the right sample chamber side.
4. Connect the RS232 port of the APG with the port labeled "ACC2" on the right side of the SPECORD PLUS.
5. Insert the sample tray.

   Note the positioning of the tray. The label has to be legible if you face the front of the device. The two centering pins (black plastic) on the contact surface of the APG protrude into the drill holes in the tray floor.
6. Clamp the stainless steel cannula loosely into the left opening of the cannula holder. The cannula is used to guide and stabilize the sample intake hose.

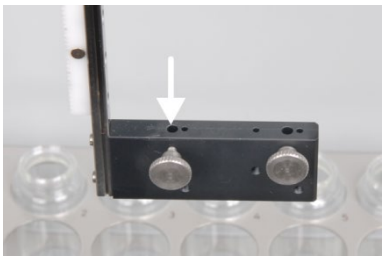


**Fig. 82 Position of the cannula in the universal cannula holder**

7. Thread the sample intake hose through the cannula so that the end protrudes from the cannula, and adjust the length of the intake hose:
   - Place a sample container into the tray.
   - With the **APG switched off (!)** guide the sampler arm with the cannula over the sample container and move the cannula holder down against the stop.
   - The stainless steel cannula must not dip into the sample. If necessary, move the cannula upwards in the holder and clamp the cannula with the knurled head screw in this position.
   - Move the intake hose down until it is approx. 1–2 mm above the container floor.

   If a magnetic stirrer is used: Place the magnetic stirrer in the sample container. Adjust the immersion depth such that the intake hose does not touch the stirrer magnet.
8. Finally connect the mains cable to the power supply and connect it to the mains socket.

9. Switch the APG on by the device switch.

   ✓ The green control LED at the front panel illuminates. The APG is initialized. All drives are moved into their start positions. The interface is now ready to receive the commands from the SPECORD PLUS.

Switching on sequence

The APG is detected during the initialization of the SPECORD PLUS and displayed in the method parameters of ASpect UV.

Always switch on the devices in the following order:

1. Switch on the APG.
2. Switch on the PC and the SPECORD PLUS.
3. Start ASpect UV and initialize the SPECORD PLUS.

**Attention**

Always observe this order when switching on to align the sampler in the correct start position.

If the sampler was switched off, re-start the software after switching the sampler back on.

Moving to individual positions

The sampler is capable of being moved to individual positions separately.

1. In the main window, select INSTRUMENT ▸ ACCESSORY ▸ SAMPLE POSITION.
2. In the APG XYZ AUTOSAMPLER window, use the buttons to move the sampler to the desired position.

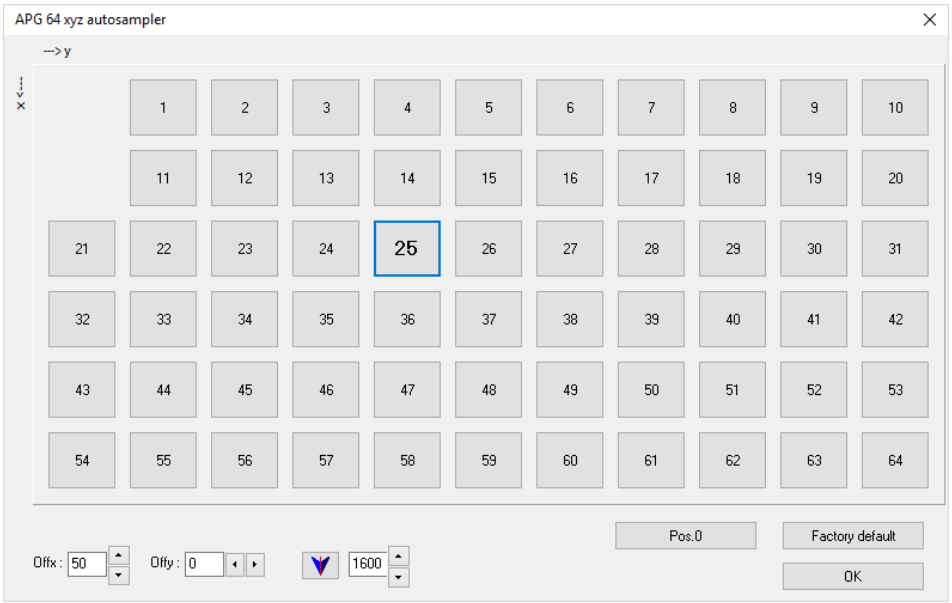



**Fig. 83 APG XYZ AUTOSAMPLER window**

## 19.2 Adjusting the sampler

The stainless steel cannula of the sampler has been adjusted so that it dips into the middle of the sample tubes (Factory default). If different sample tubes are used, the cannula of the sampler needs to be readjusted:

1. Fill the sampler with a sample tube and move to this position.
2. Using the arrow keys in the APG XYZ AUTOSAMPLER window, align the cannula with the cup:

| Parameter / button | Description |
|---|---|
| OFF X / OFF Y | Adjust the sample aspiration hose so that it is over the center of the sample tube. |
|  | Adjust the lift of the sampler arm.<br>**Note:** For the final immersion depth, move the sample aspiration hose with the sample arm lowered. |
| FACTORY DEFAULT | Restore the factory settings |

| APG steps | Description |
|---|---|
| minimum (1 step ≙) | Δx = 0.297 mm<br>Δy = 0.25 mm<br>Δz = 0.0785 mm |
| maximum | Δx = 160 mm<br>Δy = 342 mm<br>Δz = 145 mm |

For the optionally available sampler APG S, ASpect UV additionally offers the option of adjusting the sample tray to the customers' requirements.

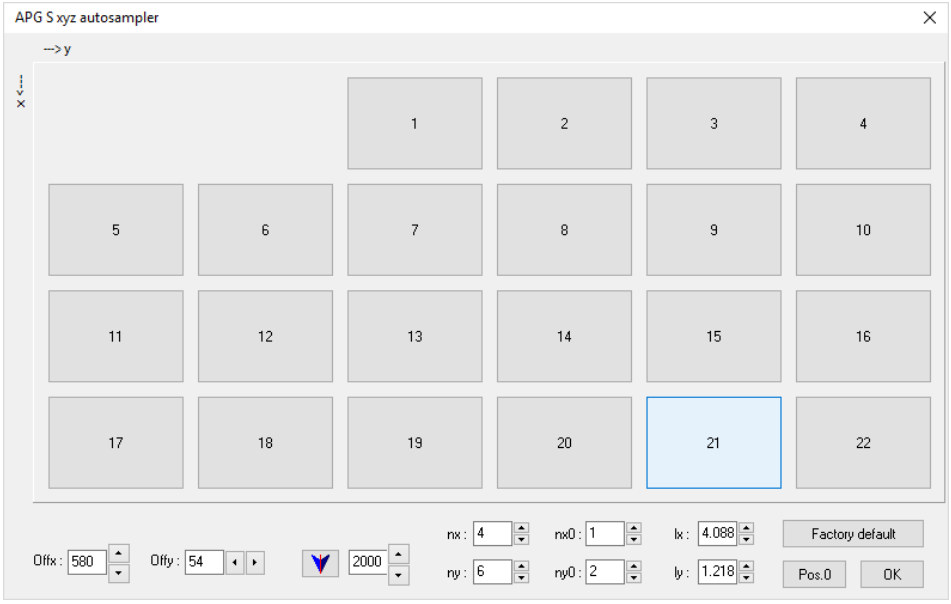



**Fig. 84 APG S XYZ AUTOSAMPLER window**

| Parameter / button | Description |
|---|---|
| NX / NY | Set the number of sample tubes via the number of table rows (nx) and columns (ny). |
| NX0 / NY0 | Shift position 1 in x/y direction |
| LX / LY | Set the sample position via the spacing of the sample tubes relative to each other.<br>**Note:** The graphic representation in ASpect UV is not adjusted. |

Optimize pump time

The pump time of the cassette sipper system must be optimized for the transport path of the sampler.

1. Provide reference and sample solution.
2. Start the optimization of the pumping time by choosing INSTRUMENT ▸ ACCESSORY ▸ OPTIMIZE PUMP TIME.
3. Click on SAMPLE POSITION to open the APG S XYZ AUTOSAMPLER window and move to the position of the reference.
4. Continue as described for the pump time optimization of the cassette sipper system (→ "Adjusting the flow cell and determining the pumping time" p. 93). Use the button to move to the position of the sample.

## 19.3 Measuring with APG and the cassette sipper system

Configurations in the measuring parameters

Make the following settings under Method on the ACCESSORY tab:

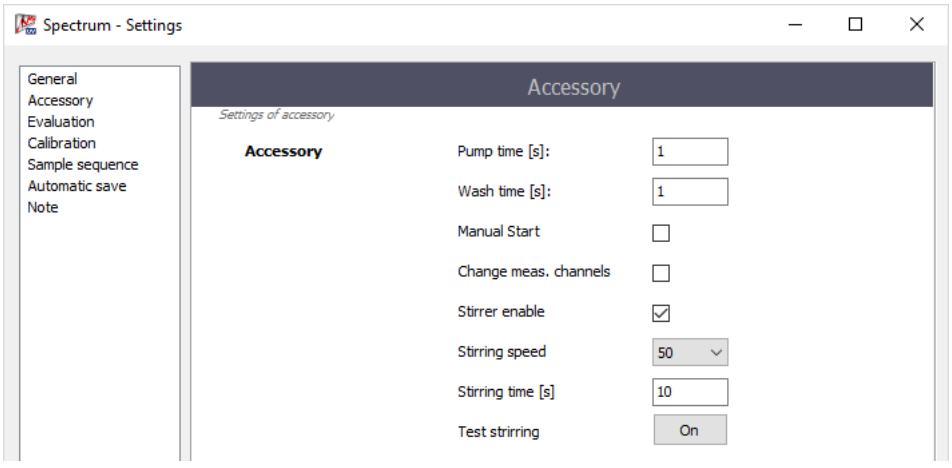



**Fig. 85 Measuring parameter configurations for the sampler**

| Parameter / button | Description |
|---|---|
| PUMPING TIME [S] | Time for transporting the sample into the cell<br>Enter the pumping time determined. |
| WASH TIME [S] | Time for aspiration of the rinsing liquid |

| | |
|---|---|
| MANUAL START | Start measuring a sample only after a further press of a button.<br>This option must be enabled if multiple measurements are run per sample. |
| CHANGE MEAS. CHANNELS | Swap the function of the beam paths in the sample chamber (measuring and reference). |
| ENABLE STIRRER | For samplers with stirrer: Activate the stirring function. |
| STIRRING SPEED | Set the stirring speed in percent.<br>The maximum stirring speed corresponds to 100 %. |
| STIRRING TIME | Set the stirring time for the sample before the sample is aspirated and transported to the cell. |
| TEST STIRRING FUNCTION | Switch stirrer on and off to determine the optimum stirring speed. |

Cassette sipper system and APG can be controlled independently of the measurement via the buttons in the menu bar:

| Button | Description |
|---|---|
|  <br>RUN SIPPER/STOP SIPPER | Switch the pump on and off.<br>This function can be used to purge the system additionally.<br>Note: At the end of a sequence, rinse the system for a few minutes to remove any sample residue. |
|  NEEDLE DOWN /UP | Lower the cannula of the sampler into the sample and raise it again. |
|  <br>RUN / STOP STIRRER | Switch the magnetic stirrer on and off. |

Carrying out the sample measurement

1. Populate the sample tray with the samples and place it onto the sampler.
2. Specify a sequence with reference and sample measurements. Use the **[W]** button to specify rinse samples.
3. Start measurement with  . The measurement starts immediately. The reference and sample measurements as well as the rinse steps are carried out as per the entries in the sequence.

## 19.4 Care and maintenance

The maintenance for the APG is limited to replacing the hoses. Pump hoses made of different materials or with different inside hose diameters are available from our customer service.

Please observe the following:

- Avoid contamination of APG and cassette sipper system. Wipe spilled samples or reagents immediately with an absorbent cloth or piece of paper. Remove

stubborn contamination with a soft cloth wetted with a commercial dishwashing detergent.

- After completing the work fill the flow cell with distilled water. If the cell dries out completely any sample residue might get stuck in the flow cell.
- After completing the work, detach the hose cassette from the pump in order to relieve the pump hoses. This ensures that the pump hoses remain elastic for longer.
- Before removing APG and cassette sipper system from the sample chamber, always switch it off via software control (INSTRUMENT ▸ ACCESSORY ▸ ACCESSORY OFF) or switch the SPECORD PLUS off by the mains switch. The accessory can then be removed from the sample chamber without risk of short circuit.

# 20 Dissolution applications

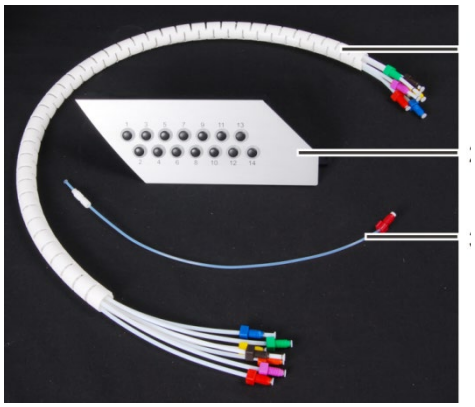
SPECORD PLUS Dissolution are automatic test systems for UV VIS measurements to analyze the dissolution behavior and release of active ingredients online. The variable equipment with one or two 8-cell changers allows for the connection of all online Dissolution systems.

Control of the spectrophotometer takes place via the software of the Dissolution system provider.

Hose routing adapters for the side walls are required to connect to a Dissolution system. When two 8-cell changers are used, up to 14 sample channels can be connected. The appropriate hoses and cells can be purchased from the provider of the Dissolution Test system.


**Fig. 86 The SPECORD PLUS with connected Dissolution system**

Side components and hose connections



1 Hose connections between Dissolution system and SPECORD PLUS
2 Side components with hose adapters
3 Hose coupling between side component and flow cell
4 Black foam component for the suppression of extraneous light

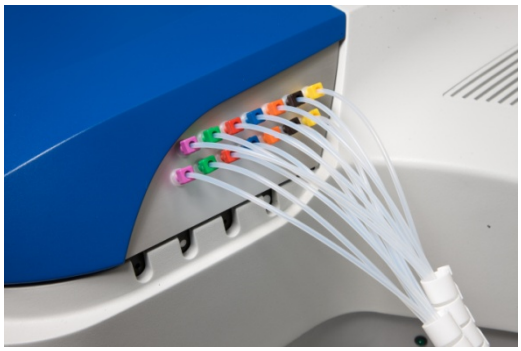

**Fig. 87 Side components and connection hoses**

The side components each have 14 numbered hose adapters sealed light-proof with plugs. The hose connections to the Dissolution system are bundled. The color coding facilitates the assignment of the sample containers to the flow cells.

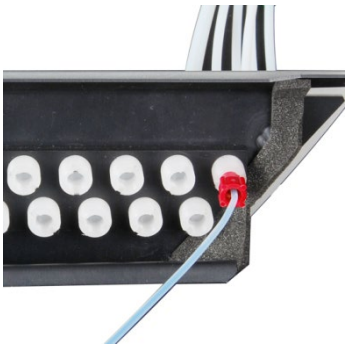
The hoses between the side component and the flow cells have two different connections: a colored one to connect to the side components and a PTFE connection to the flow cell. The hoses are fitted with two PTFE connections of different lengths. The shorter one is screwed into the inlet channel of the cell, the longer one into the outlet channel.

Connecting the Dissolution system to the SPECORD PLUS

1. If the cell changers are used: Install the cell changer in the sample chamber as described in section "8-cell changer" p. 41.
2. Replace the side components of the SPECORD PLUS by side components with hose adapters.
3. Remove the black plugs from the openings of the adapters.
4. Screw the PTFE hoses for the supply and removal of the samples to the outsides of the side components. The numbering on the side components and the color codes facilitate the assignment of the sample hoses to the cell positions.
5. Screw the hoses with a short PTFE connection to the inside of the side component to which the sample is supplied.
6. Screw the hoses with a long PTFE connection to the inside of the side component from which the sample flows back to the Dissolution system.
7. Thread the hoses on both sides inside through the pre-punched holes in the foam components (4 in Fig. 87). Move the foam components for the suppression of extraneous light close to the side walls.
8. Screw the short PTFE connections to the inlet apertures of the flow cells. Screw the long PTFE connections to the outlets of the flow cells. The flow direction is marked on the cells.
9. Position the flow cells in the cell changer.
10. Use cable ties to tie the hoses in the sample chamber together, aligning them so that they do not protrude into the beam path.



Side component with connected hoses on the outside (to/from the Dissolution system)



Side components with connected hoses on the inside (to/from the flow cell)


Flow cell

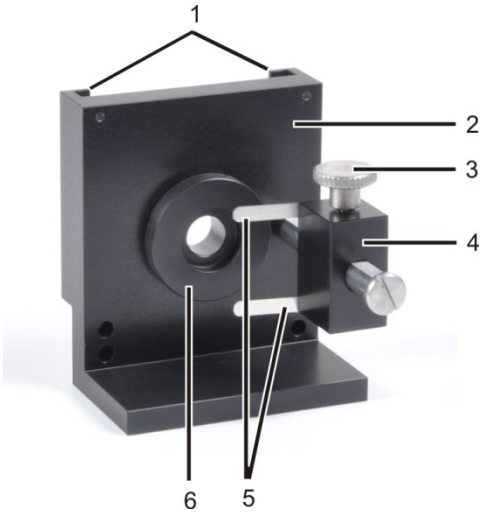
**Fig. 88 Hose connections for connecting a Dissolution system**

# 21 Holder for solid samples

The holder is suitable for transparent solid samples with diameters from 17 mm upwards and a pathlength of up to 25 mm. The maximum sample dimension can be 80 mm x 140 mm.

The holder is equipped with an adapter sheath which makes it possible to measure samples with a diameter from 9 mm upwards. If the geometry of the sample permits it, the adapter sheath should also be used for larger samples. The adapter sheath prevents refraction and scatter losses that could distort the measurement results.

Layout




1 Guide
2 Contact surface for the sample
3 Knurled head screw
4 Support block
5 Contact spring
6 Adapter sheath

**Fig. 89 Holder for solid samples**

Installing the holder and inserting the sample

1. Slide the holder with the guide onto the adapter plate in the front sample chamber wall in the measuring channel.
2. Loosen the knurled head screw and pull back the support block.
3. Place the sample onto the contact surface of the holder.
4. Slide the support block against the sample until the contact springs securely hold the sample.
5. Tighten the knurled head screw.

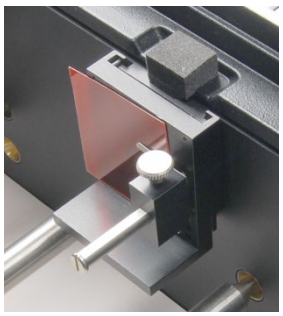


**Fig. 90 Holder for solid samples installed in the SPECORD PLUS**

# 22 Absolute reflectance attachment

With this accessory it is possible to determine the absolute reflectance of reflecting layers that are homogeneous and bright polished and have a constant thickness.

| | |
|---|---|
| min. sample size | 34 x 34 $mm^2$ (or Ø = 34 mm) |
| max. sample size | 130 x 130 $mm^2$ |
| Reflection angle | 7° |

Optionally, adapters for smaller samples with the diameters of 20, 25 and 25.4 mm are available. The smallest possible sample diameter is 18 mm. They accommodate samples of a thickness of 6 mm and are inserted into the contact plate.

The absolute reflectance attachment is equipped with an identification connector. This connector ensures that the attachment is detected during device initialization and enabled in the method parameters.

Layout

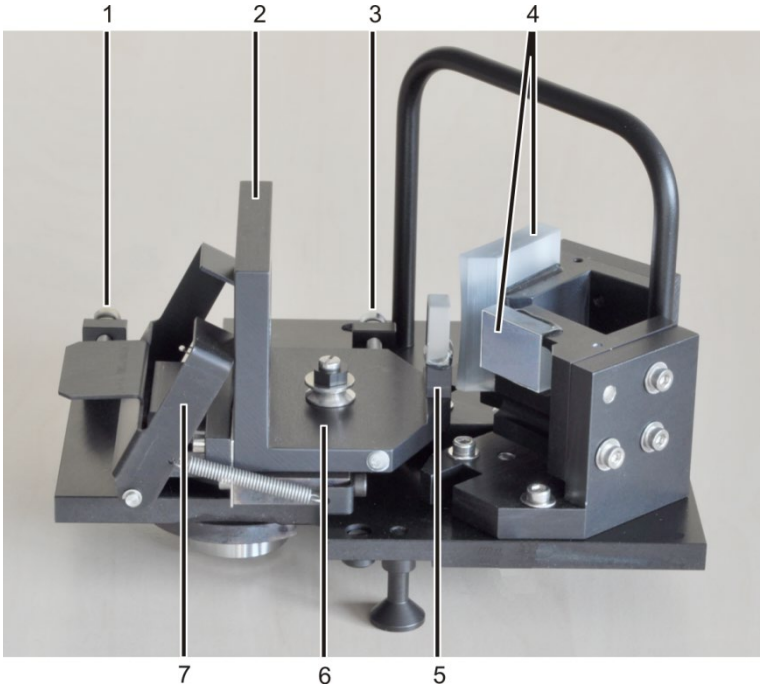



**Fig. 91 Absolute reflectance attachment**

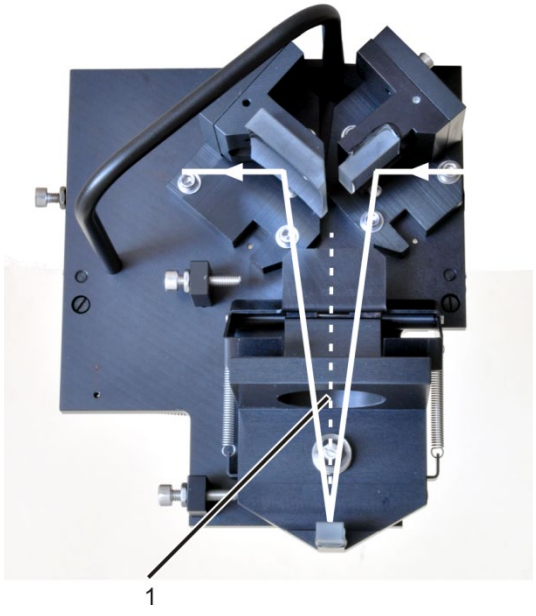
1 Stop bolt for the V-shaped beam path
2 Contact plate for the sample
3 Stop bolt for the W-shaped beam path
4 Fixed mirror
5 Pivotable mirror
6 Frame for pivotable mirror
7 Sample holder

Principle of operation

The reflectance is determined in two steps:

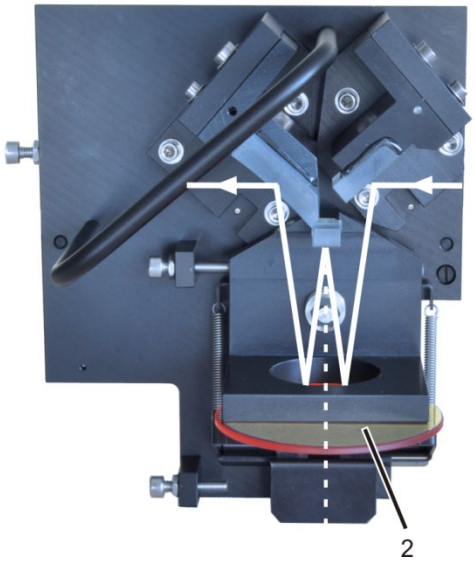
1. Determination of the reference in the V-shaped beam path
2. Measuring the sample in the W-shaped beam path

The measurements are carried out with a reflection angle of 7°.




V-shaped beam path for reference measurement

The reflection takes place at the mirror, rotating mirror and second mirror.




W-shaped beam path for sample measurement

The pivotable mirror is rotated by 180°. The sample is at the pivoting point where the beams are reflected twice. The other reflections (mirror/rotating mirror/mirror) are identical to the reference measurement.

**Fig. 92 V- and W-shaped beam paths at the attachment**

1 Pivoting point of the pivotable mirror
2 Sample

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

$$R = \frac{r_{Probe}}{r_{Referenz}}$$

$r_{sample}$ – sample measurement in W position
$r_{reference}$ - reference measurement in V position

$$r_{Referenz} = \frac{I_{RS}}{I_{Vergleichsstrahl}}$$

$I_{RS}$ – intensity after reflection at the reference mirror
$I_{reference\ beam}$ – intensity in the reference beam path

$$r_{Probe} = \frac{I_{PS1} \times I_{RS} \times I_{PS2}}{I_{Vergleichsstrahl}}$$

$I_{PS1}$ – intensity after the first reflection at the sample
$I_{PSI2}$ –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, $I_{PS1} = I_{PS2}$ applies. This results in the absolute reflectance of the sample $R_{abs}$:

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

The value $R_{abs}$ is displayed in the graphical display or the measurement table in percentage values.

## 22.1 Installing the attachment in the sample chamber

### Attention

Danger of damage to the sensitive optical mirrors!

Work with care and avoid the contamination of the mirrors.

Do not touch the mirrors!

1. Screw the support rods into the top position (→ "Converting the sample chamber" p. 9).
2. Before mounting the attachment, remove the left support rod from the reference channel.

   **Note:** The support rod hinders the installation. Once the attachment is installed, the support rod can be mounted again.
3. Place the attachment onto the support rods in the measuring channel. Slide the attachment up to the stop against the front sample chamber wall.
4. Press down the base plate until it engages on the support rods with a click.
5. Connect the identification connector to the connection (**M**) in the right sample chamber wall.

   ✓ The attachment is installed in the SPECORD PLUS.

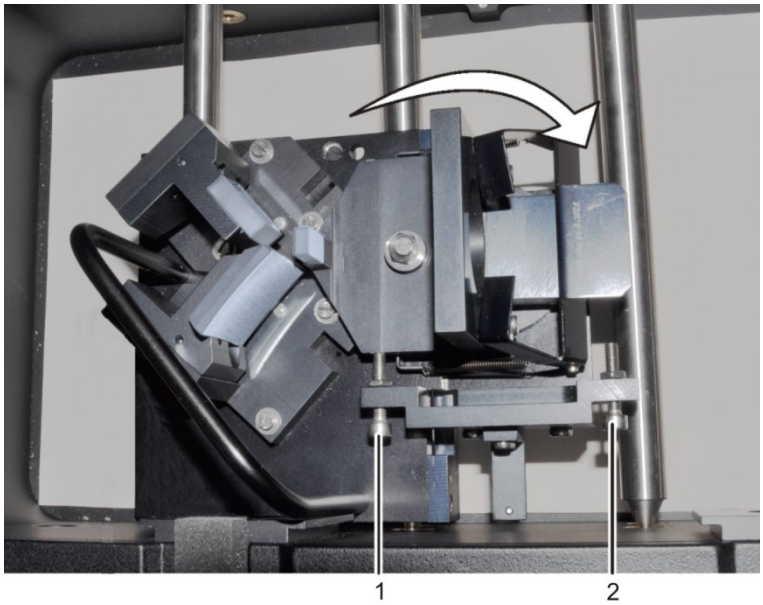



**Fig. 93 Absolute reflectance attachment in the sample chamber**

1 Stop bolt for the W-shaped beam path 2 Stop bolt for the V-shaped beam path

## 22.2 Adjusting the attachment

If the attachment and the SPECORD PLUS were delivered together, the attachment is adjusted and can be used without further adjustment.

If an attachment that was delivered subsequently is used, the adjustment of this attachment needs to be checked.

1. Install the attachment into the sample chamber, without connecting the identification connector to the interface in the sample chamber.
2. Configure the zero order of the Vis lamp (→ see section "Configuring the zeroth order" p. 11).
3. Tilt the attachment into the V position.
4. Adjust the holder by moving the stop bolt (2 in Fig. 93) until the light beam passes through the center of the measuring position for cloudy samples and thus hits the center of the receiver. To observe the beam path place a white screen, e.g. a strip of paper, into the cell measuring position for cloudy samples.
5. Tilt the attachment into the W position.
6. Adjust the holder by adjusting the stop bolt (1 in Fig. 93) as described in item (4).
7. In the PHOTOMETRY module set a method with the following parameters:

   GENERAL tab:

| Parameter | |
|---|---|
| MEAS. MODE | TRANSMITTANCE |
| WAVELENGTHS [NM] | 500.00 |
| INTEGRATION TIME [S] | 0.1 |
| SLIT [nm]<br>(SPECORD 210/250 PLUS only) | 1 |

   SAMPLE SEQUENCE tab:

   - One reference measurement in first position
   - Further sample rows with the sample type SAMPLE for adjustment

8. Start the measurement with  .
9. First change the adjustment in the V position.Measure the transmission in each case **with the sample chamber cover closed**. Repeat the process until the transmittance value reaches its maximum.
10. Do the same in the W position, using a plane mirror as sample.

    ✓ The attachment has been adjusted.

11. Connect the identification connector to the connection (**M**) in the right sample chamber wall.

## 22.3 Measurements with the attachment

The absolute reflectance attachment is automatically detected during the device initialization in ASpect UV and displayed in the method parameters.

In all modules only the **Reflection** option is available as **measuring mode**. Configure all other method parameters for the specific measuring task.

Perform measurements as follows:

1. Specify the reference and sample measurement in the sequence.
2. Start the measurement by clicking on  and follow the instructions on the screen.
3. Tilt the attachment into the V position and start the reflection measurement.
4. Tilt the attachment into the W position.
5. Place the sample with the reflective surface against the contact surface and carefully clamp the samples with the sample holder.
6. Start the sample measurement.
   ✓ The absolute reflectance is displayed as the measuring result.

## 22.4 Care and maintenance

### Attention

Danger of damage to the sensitive optical mirrors!

Work with care and avoid the contamination of the mirrors.

Do not touch the mirrors!

Please observe the following maintenance instructions:

- Keep all mirrors of the attachment free from dust and grease! Do not touch the mirrors with your fingers!
- Remove dust particles on the mirrors with a soft, clean and grease-free brush.
- To remove traces of grease on the mirrors: Carefully and without applying pressure wipe the mirrors with a cotton pad soaked in distilled water and curd soap.
  Wipe the mirrors with distilled water from a wash bottle. Dab the metal frames dry afterwards.
- Only transport and store the attachment in the sealed container.

# 23 Reflection measuring attachment 11° – 60°

The attachment is used to determine the reflectance of a solid sample at a variable reflection angle of 11° – 60°.

The reflection measuring attachment can be used to determine the pathlength and refractive index of a solid sample. For this purpose, reflectance measurements with different reflectance angles are performed in a defined wavelength range. To determine the pathlength the interferences occurring during the measurement are used. The existing number of maximum interference values in a defined wavelength range is analyzed. Prior to the sample measurement a reference measurement must be carried out where the mirror supplied must be used instead of the sample.

Technical data

| | |
|---|---|
| Reflection angle | 11° – 60° |
| Adjustable part interval of the angular scale | 1° |
| Vignetting of the beam bundle | In the angle range 11°-15° |
| Minimum size of the sample surface | 12 x 10 $mm^2$ |
| Maximum sample thickness | 30 mm |
| Illuminated sample surface | 2.5 x 6 to 2.5 x 12 $mm^2$ dependent on the reflection angle set |
| reference sample | Mirror with aluminum surface and protective coating |
| Dimensions | 165 x 115 x 135 $mm^3$ |
| Dimensions of the table top for small samples | 115 x 80 $mm^2$ |
| Dimensions of the small table top for large samples | 70 x 80 $mm^2$ |
| Mass | 2 kg |

Layout and function

The measuring attachment features a base plate (10 in Fig. 94), on which the imaging and reflection optics and a sample table are mounted.

The sample table can be pivoted horizontally for **reflection angles in the range of 11° – 60°**. The wavelength of the measuring beam is identical for all adjustable angles.

A table top for smaller samples or a table for larger samples can be slid onto the table. The samples can be secured on the table tops. The table top for smaller samples sits unmovably on the table during the measurement and can be locked by means of a knurled head screw. Markings on it are used to relocate the sample position. With the table top for larger samples, samples can be moved on the table and measured step by step, e.g. to check their homogeneity.

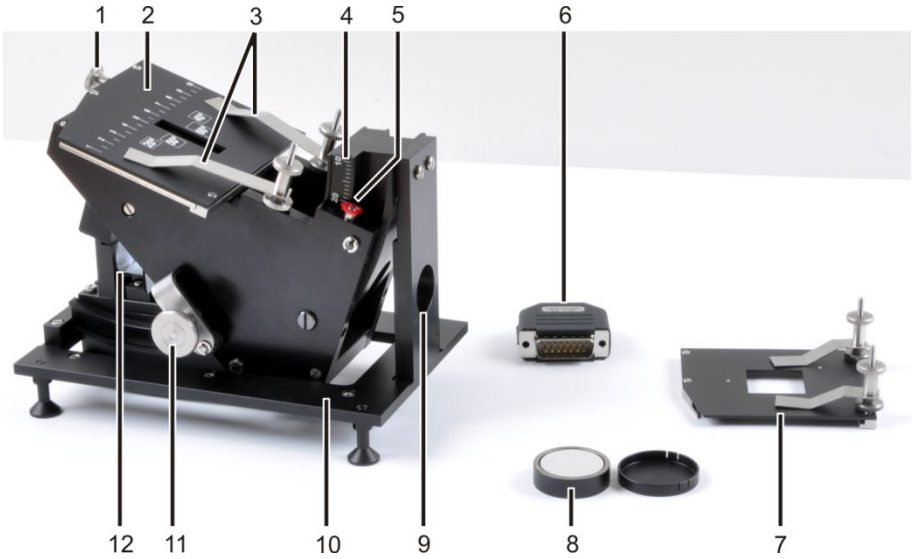
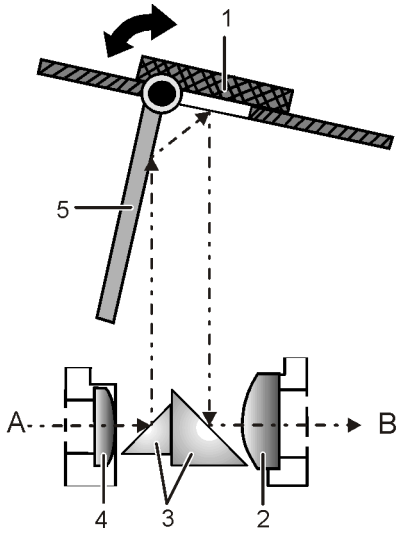



**Fig. 94 Reflection measuring attachment**

1 Knurled head screw
2 Table top for small samples
3 Sample contact springs
4 Adjustment scale for reflection angle
5 Pointer for set reflection angle
6 Identification connector
7 Table top for large samples
8 Reference mirror (with lid)
9 Beam outlet
10 Base plate
11 Knurled head screw to fix the reflection mirror
12 Beam entry




1 Sample
2, 4 Quartz lenses
3 Deflection mirror
5 Plane mirror
A Beam entry
B Beam outlet

**Fig. 95 Beam path in the reflection measuring attachment (viewed from the front)**

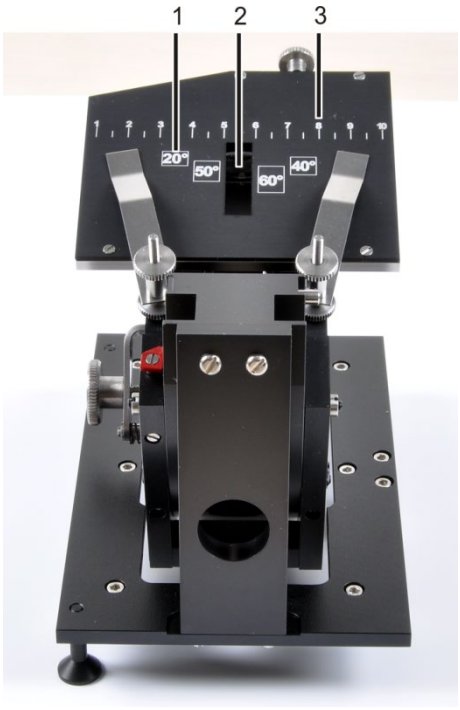



Table top for small samples

1 Marking for illuminated sample surface
2 Measuring aperture
3 Scale for reproducible positioning of the sample

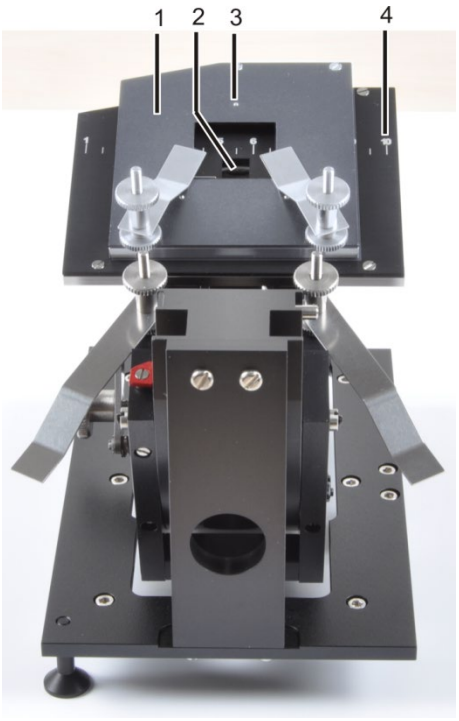



Table top for large samples

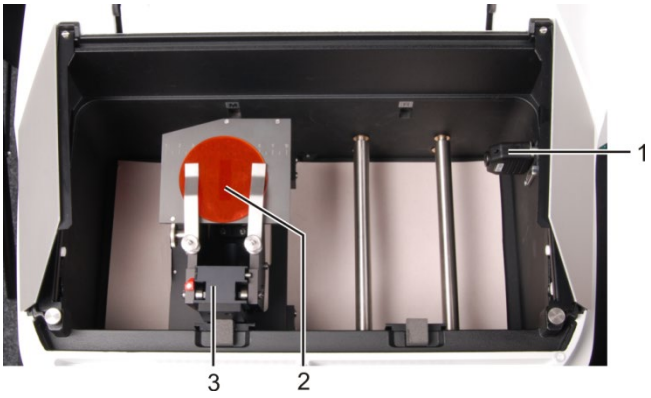
1 Movable table top
2 Measuring aperture
3 Point support to protect the sample surface against scratching
4 Scale on the table for reproducible positioning of the sample

**Fig. 96 Table tops for reflection measuring attachment**

## 23.1 Installing the reflection measuring attachment into the sample chamber

1. Screw the support rods in the sample chamber into the top position (→ "Converting the sample chamber" p. 9).
2. Place the attachment onto the support rods in the measuring channel. The adjustment scale for the reflection angle points to the left. Slide the attachment against the front sample chamber wall.
3. Press down the base plate until the attachment engages on the support rods with a click.
4. Connect the identification connector to the connection (**M**) in the right sample chamber wall.

   ✓ The attachment is installed in the SPECORD PLUS.




1 Identification connector
2 Sample
3 Reflection measuring attachment

**Fig. 97 Reflection measuring attachment installed in the sample chamber**

Setting the reflection angle

1. Loosen the knurled head screw (11 in Fig. 94).
2. Tilt the sample table into the desired angle position.
3. Tighten the knurled head screw.

**Note:** The beam bundle is vignetted in the angle range of 11° – 15°. The signal/noise ratio of the measurement deteriorates:

## 23.2 Measurements with the reflection measuring attachment

The reflection measuring attachment is automatically detected during the device initialization in ASpect UV and displayed in the method parameters. In all modules only the REFLECTION option is available as MEASURING MODE. Adapt all other method parameters to your individual measuring task.

1. Specify the reference and sample measurement in the sequence.
2. Place the reference mirror supplied as a reference on the table top and clamp it with the contact springs.
3. Start the sequence by clicking on ▶, and record the reference values.
4. Replace the reference mirror with the sample. Start the sample measurement.

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

$$R = \frac{r_{Probe}}{r_{Referenz}}$$

$$r_{Referenz} = \frac{I_{RS}}{I_{Vergleichsstrahl}}$$

$$r_{Probe} = \frac{I_{PS}}{I_{Vergleichsstrahl}}$$

$r_{sample}$ – sample measurement

$r_{reference}$ reference measurement

$I_{RS}$ – intensity after reflection at the reference mirror

$I_{reference\ beam}$ – intensity in the reference beam path

$I_{PS}$ – intensity after reflection at the sample

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

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

✓ The value $R_{rel}$ is displayed in the graphical display or the measurement table in percentage values.

Calculation of the pathlength and refractive index

Assuming that the refractive index is the same for all wavelengths, the pathlength and refractive index can be calculated using the method described below. The pathlength of a sample with a known refractive index can be calculated using the following formula:

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

| | |
|---|---|
| d: | Sample thickness |
| m: | Number of maximum interference values after zeroed maximum (see example) |
| n: | Refractive index of the sample |
| $\Theta$: | Reflection angle set |
| $\lambda_1$: | Wavelength of the zero-th interference maximum |
| $\lambda_2$: | Wavelength of the m-th interference maximum |

Example: Measuring curve (recorded at a reflection angle of 60°) with 5 maximum interference values. The first maximum value is not counted.

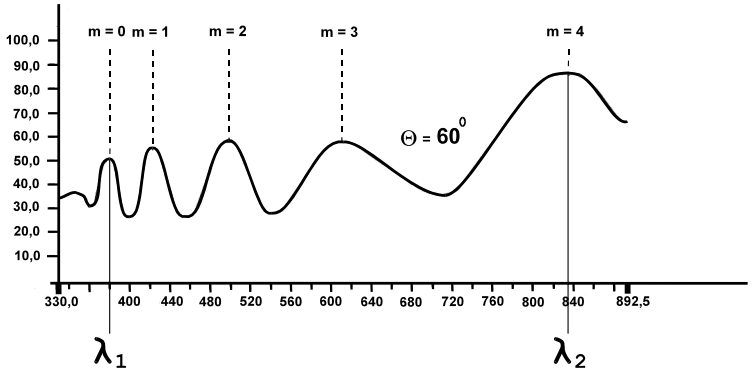



**Fig. 98 Example interference values at a reflection angle of $\Theta$ = 60°**

The wavelengths $\lambda_1$ and $\lambda_2$ must be determined as accurately as possible. To do so, in the ASpect UV Software in the SPECTRUM module, use the EVALUATION ▸ PEAKS menu command.

To calculate the refractive index (with unknown sample pathlength) measurements must be carried out with at least two different reflection angles, because the formula includes two unknowns (d, n). The calculation is then carried out on the basis of the solution of an equation with two unknowns.

**Note:** For the most accurate determination of the refractive index it is recommended to carry out measurements at several reflection angles and take the arithmetic mean of the results.

## 23.3 Care and maintenance

**Attention**

Danger of damage to the sensitive optical mirrors!

Work with care and avoid the contamination of the mirrors.

Do not touch the mirrors!

Please observe the following maintenance instructions:

- Keep all mirrors of the attachment free from dust and grease! Do not touch the mirrors with your fingers!
- Remove dust particles on the mirrors with a soft, clean and grease-free brush.
- To remove traces of grease on the mirrors: Carefully and without applying pressure wipe the mirrors with a cotton pad soaked in distilled water and curd soap.
  Wipe the mirrors with distilled water from a wash bottle. Dab the metal frames dry afterwards.
- Only transport and store the attachment in the sealed container.

# 24 Integrating sphere

The integrating sphere is used for the measurement of scattering solid or liquid samples in transmittance or reflectance.

In reflectance the sphere operates with the measuring geometry 8°/d.

Note that the sphere reduces the energy of the measuring signal compared to the beam path without sphere to a few percentage points. This means that measurements with great accuracy can only be carried out up to an absorption of 2.

The integrating sphere has a spherical diameter of 75 mm and consists of two Spectralon hemispheres with apertures for the beam entrance and exit, beam deflecting optics and holders for samples and cells for transmittance and reflectance measurements.

The integrating sphere is inserted into the measuring channel of the sample chamber. To populate it with powdered samples it can be removed from the sample chamber and reinserted reproducibly.

Technical data

| | |
|---|---|
| Sphere material | Spectralon |
| Sphere diameter | 75 mm |
| Light–Entrance aperture | ∅ 10 mm |
| Reflectance aperture | ∅ 12 mm |
| Light–Exit aperture | ∅ 16 mm |
| Transmittance cells | Width 12.5 mm<br>Pathlength up to 10 mm |
| Solid transmittance samples | Length up to 250 mm<br>Width up to 85 mm |
| Reflectance samples | Diameter up to 50 mm<br>Thickness up to 35 mm |
| Adapter for powdered samples | Diameter 16 mm<br>Depth 5 mm |
| Dimensions of the spherical body ( L x W x H ): | 150 mm x 105 mm x 145 mm |
| Weight | 1.0 kg |

Layout and function

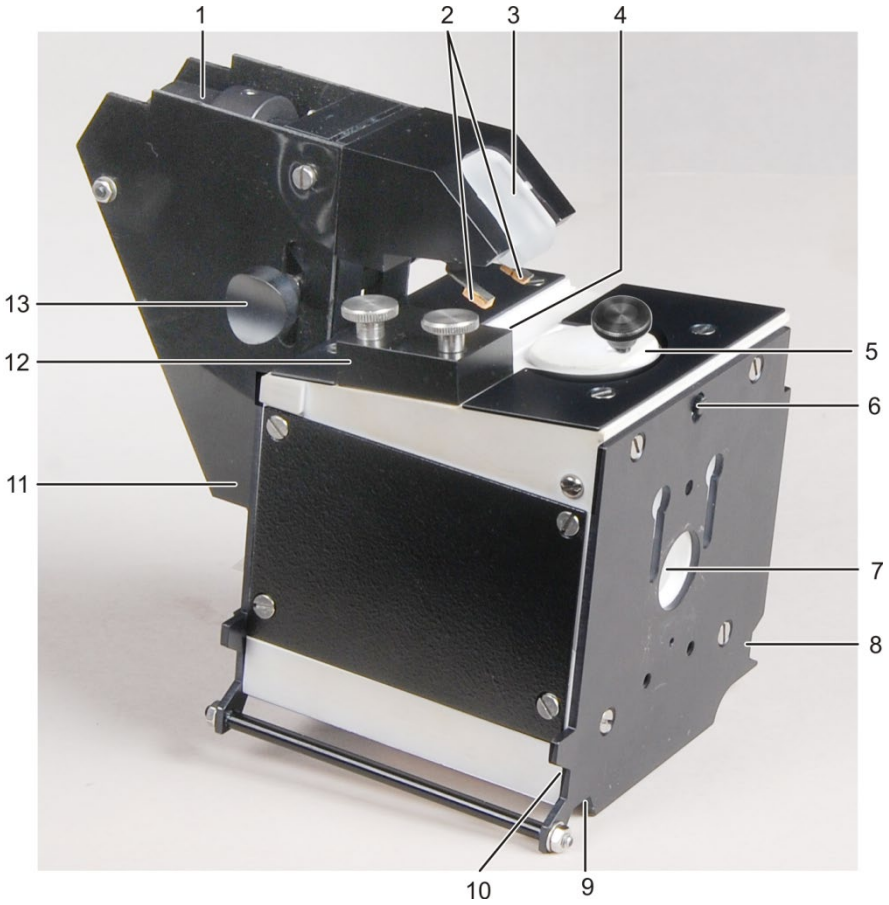



**Fig. 99 Layout of the integrating sphere – View of the transmittance aperture**

1 Mirror 2
2 Retention springs for transmittance samples
3 Mirror 3
4 Beam entrance aperture
5 Aperture for gloss trap with Spectralon closure
6 Dowel pin with knurl
7 Beam outlet aperture to the detector
8 , 9, 10 Supports on the support rods for vertical and horizontal installation
11 Mirror 1
12 Contact surface for cells
13 Adjustment handle for the retention springs

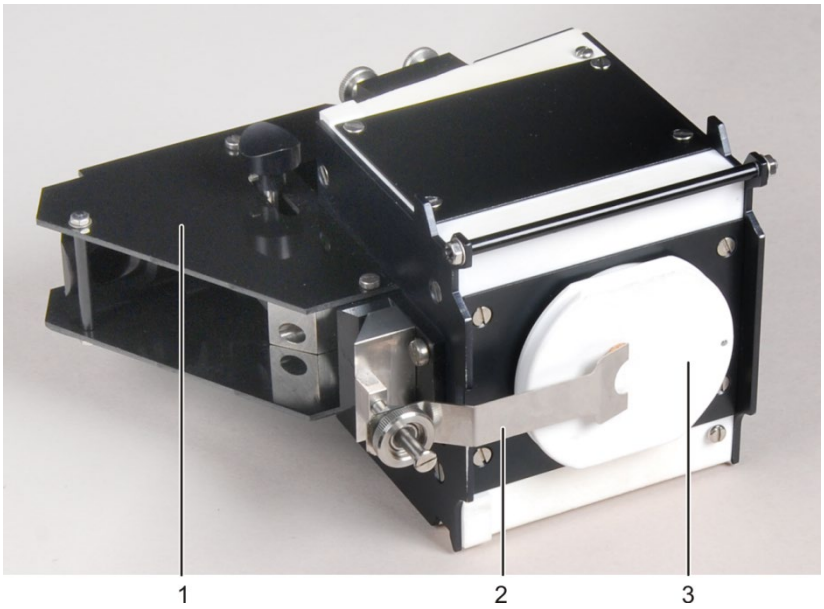



**Fig. 100 Layout of the integrating sphere – View of the remittance aperture**

1 Beam deflecting optics
2 Retention spring
3 Spectralon insert before the aperture for reflectance measurements

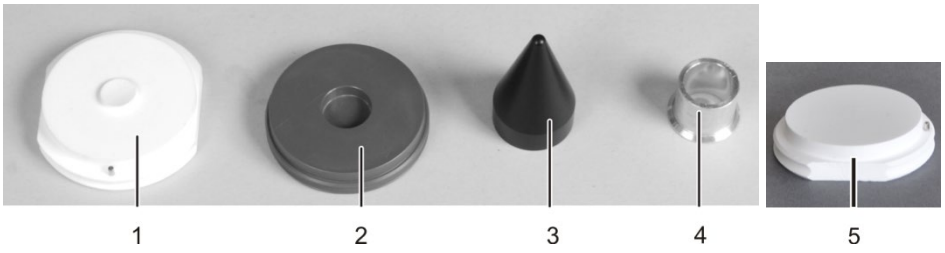



**Fig. 101 Accessories of the integrating sphere**

1 Spectralon insert to close the reflectance opening for transmission measurements
2 Sample carrier for powdered samples
3 Gloss trap
4 Reflector
5 Spectralon insert as a reference for reflectance measurements

The integrating sphere is placed onto the sample chamber rods in the sample chamber. The spherical body can be used in two different positions in the sample chamber dependent on the measuring type and sample requirements:

- Vertical:
- Horizontal:

Vertical installation

The beam deflecting optics is vertical. The beam inlet aperture is at the top and the reflectance sample below the spherical body. This position can be used for the transmittance measurements of solid bodies and reflectance measurements of powdered samples.

Horizontal installation

The beam deflecting optics is in a horizontal position. The beam enters the sphere from the side, the aperture for reflectance is accessible from the side. This position can be used for the transmittance measurements of samples in cells and reflectance measurements of compact samples.

During transmittance or reflectance measurements the sample is moved directly to the sphere. It thus forms part of the inside surface of the sphere and affects the sphere efficiency. In the case of transmittance the radiation is e.g. weakened by the absorption in the sample. However, because the sample itself is also part of the sphere, the sphere efficiency changes dependent on the reflection and reflectance properties of the sample, i.e. its efficiency depends on the properties of the sample. This results in a non-linear relationship between the sample absorbance and measuring signal. For high precision measurements it is therefore recommended to take this non-linear relationship into account by way of a calibration similar to a quantitative analysis. Certified standard samples should be used for the calibration.

## 24.1 Unpacking and storage

### Attention

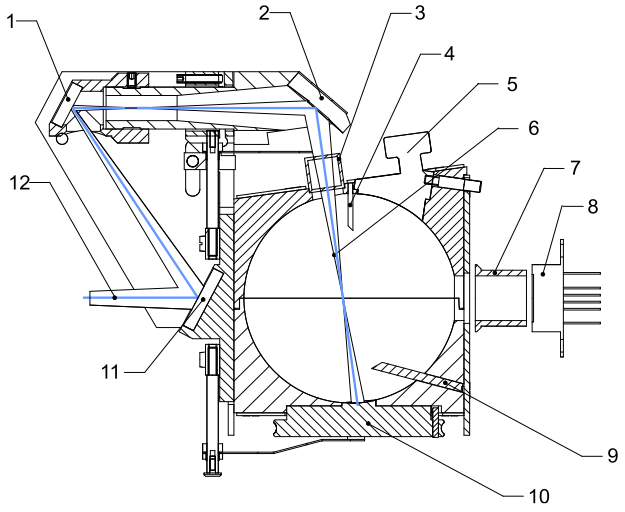
**Sensitive optical instrument**

With its open mirrors and the highly reflective Spectralon components, the integrating sphere is a very sensitive optical instrument. Especially when handling powdered samples, care must be taken to prevent powder particles or other contamination from entering the spherical body. A change of the reflectance of the Spectralon from 99 % to 98 % reduces the efficiency of the integrating sphere to half. The Spectralon inserts and the gloss trap must also be handled with the same care, because changes in the reflectance of those components also reduce the efficiency.

- Carefully remove the spherical body from the transport and storage container without touching the mirrors.
- Only hold the spherical body by the side panels.
- If the integrating sphere is not used, store it with all its accessories in the corresponding container.
- Though the mirrors have a $SiO_2$ – protective coating, but they should be protected against contamination.
- If the integrating sphere is not used, remove the reflector from the SPECORD PLUS. Otherwise its unprotected high gloss aluminum surface could be damaged.

## 24.2 Transmittance measurements

The integrating sphere ensures the uniform illumination of the receiver surface in the spectrometer, independent of any influence (scattering, deflection) on the beam by the sample. This improves the accuracy of the measuring results.




1 Mirror 2
2 Mirror 3
3 Cell with scattering sample
4 Aperture 1 (baffle)
5 Spectralon insert instead of gloss trap
6 non-scattered radiation
7 Reflector
8 Radiation receiver of the SPECORD PLUS
9 Aperture 2 (baffle)
10 Spectralon insert for transmittance measurement
11 Mirror 1
12 Beam distribution of the SPECORD PLUS

**Fig. 24-102 Beam path for transmittance measurements**

Aperture 1 prevents direct light reaching the detector from the sample. Aperture 2 prevents light that has not been scattered in the sample reaching the radiation receiver of the SPECORD PLUS after the first reflection at the Teflon insert.

If larger solid samples are to be measured in transmittance, you can unscrew the limit stop and the knurled head screw at the Teflon insert. You can then place your sample onto the available smooth surface.

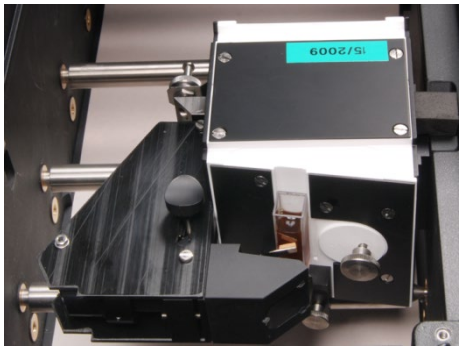
Preparation of the measurement

1. Connect the identification connector to the connection (**M**) in the sample chamber.
2. Screw the support rods in the sample chamber into the top position (→ "Converting the sample chamber" p. 9).
3. Place the reflector (4 in Fig. 101) up to the stop in the round opening in the center of the rear adapter for the cell holder.

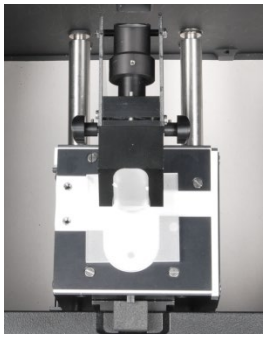
**Fig. 103 Reflector inserted into the adapter for the cell holder**

4. Insert the Spectralon insert (1 in Fig. 100) into the reflectance opening of the spherical body.
5. Insert the Spectralon closure into the aperture of the gloss trap. Bring the insert into the correct position by positioning it at the guide pin (6 in Fig. 99).
6. If required by the sample type unscrew the limit stop (12 in Fig. 99) and the knurled handle at the Spectralon insert (5 in Fig. 99).

7. Place the spherical body onto the two sample chamber rods in the measuring channel and slide it forward against the stop. During each measurement it must be placed reproducibly against the stop.
8. Install the spherical body depending on the sample type.




Horizontal installation:
Measurement of liquid samples



Vertical installation:
Measurement of solid samples

**Fig. 104 Transmittance measurements in liquid and solid samples**

Transmittance measurements in large samples

Samples with a very large surface can also be positioned at the beam exit between integration sphere and reflector. If the sample is positioned in this way, it is radiated with diffuse light. When making measurements, care must be taken to make sure that the integration sphere is at the same position during reference and sample measurements.

Carrying out a transmittance measurement

The integrating sphere is detected during the device initialization and activated in the measuring parameters.

1. In the measuring parameters select the largest possible gap to achieve a good energy level.
2. Perform the reference measurement.
3. Place the sample in the integration sphere and carry out the sample measurement.

## 24.3 Reflectance measurements

The colored appearance or the gray shade of an opaque object is due to its wavelength-dependent diffuse reflection (reflectance). Reflectance measurements can be carried out with different illumination and measuring geometries dependent on their surface structure.

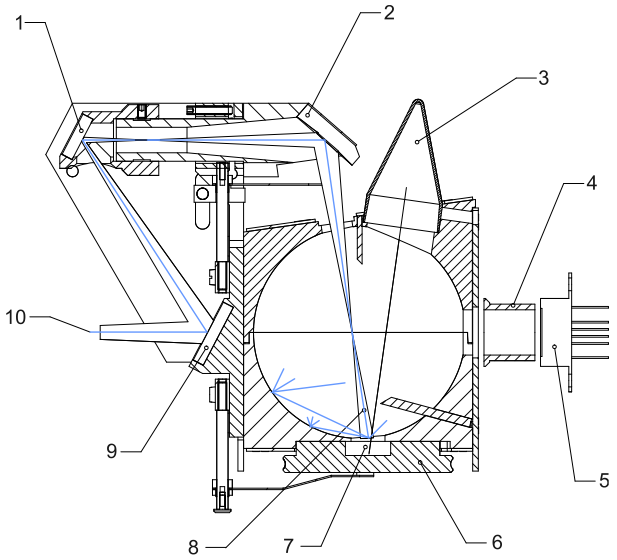
To prevent the effect of the surface structure as much as possible, the remitted radiation of the sample is captured by the integrating sphere and through the multiple reflection at the inner surface of the sphere reaches the radiation receiver of the SPECORD PLUS diffusely.

The measuring geometry of 8°/d (d = diffuse) means that the sample surface is irradiated arranged at an angle of 8 to its surface normal and the radiation reflected from the sample surface into the integrating sphere falls diffusely on the receiver.

The radiance factor of a sample is the quotient of the radiation remitted from its surface and the radiation remitted from a completely matte white surface of a

standard sample under identical optical conditions. Thus the reflectance can be related to that of a Spectralon standard sample.

The integrating sphere is preferably suited for reflectance measurements of samples with structured (rough, grained etc.) surfaces, such as cellulose, leather or textile fabrics and of samples with azimuthal gloss, i.e. a gloss that changes by the rotation of the sample around its surface normal.




1 Mirror 2
2 Mirror 3
3 Gloss trap
4 Reflector
5 Radiation receiver
6 PTFE insert for reflectance aperture
7 Reflectance aperture for reflectant powdered sample
8 Diffuse reflection
9 Mirror 1
10 Beam distribution of the SPECORD PLUS

**Fig. 105 Beam path during reflectance**

Preparation of the measurement

1. Connect the identification connector to the connection (**M**) in the sample chamber.
2. Screw the support rods into the top position (→ "Converting the sample chamber" p. 9).
3. Place the reflector (4 in Fig. 101) up to the stop in the round opening in the center of the adapter for the cell holder.
4. Reflectance measurements in powdered samples: Install the spherical body in such a way that the powdered samples are always at the bottom of the spherical body (vertical installation). In this position no powder can fall on the spherical body.

   Reflectance measurements in solid samples: Install the spherical body in such a way that the aperture for remission is at the side, making it easily accessible (horizontal installation).
5. Reflectance samples do not always scatter the incoming radiation 100%; a significant portion is also reflected as in a mirror. To eliminate this portion, use the gloss trap.
   - Remove the dowel pin (6 in Fig. 99) and pull the Spectralon insert (5 in Fig. 99) by the knurled handle out of the aperture.
   - Insert the gloss trap (3 in Fig. 101).

Preparing powdered samples

1. Fill the sample carrier with the sample (2 in Fig. 101).Lightly press down the sample and smoothen the surface.

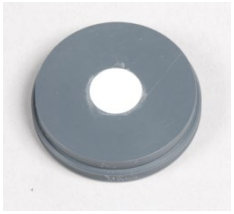


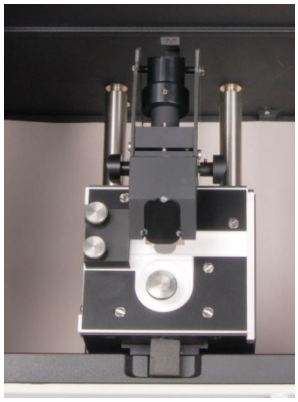
**Fig. 106 Sample carrier with powdered sample**

2. Lift the integrated sphere and attach the sample carrier from the bottom to the reflectance aperture of the spherical body.

   **Note:** When working with powdered samples or with samples that could leave abrasion marks on the spherical body made of Spectralon, use of the filter paper (included as consumable material) is recommended.

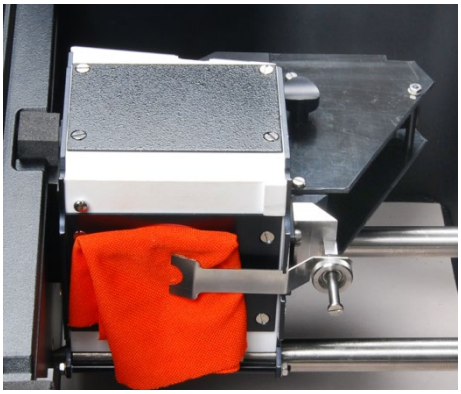
3. Both for reference and sample measurements, place the filter paper between the reflectance aperture and the sample or sample carrier, respectively.

   **Important:** Do not tilt the spherical body with the sample attached. Powder could fall into the sphere and contaminate it.



Vertical installation

Measuring of powdered samples. The sample carrier is attached from the bottom to the spherical body.



Horizontal installation

The sample can be attached from the side to the reflectance aperture.

**Fig. 107 Reflectance measurements in powders and compact samples**

Carrying out the measurements

The integrating sphere is detected during the device initialization and activated in the measuring parameters.

1. In the measuring parameters select the largest possible gap to achieve a good energy level.
2. Perform the reference measurement. Here either the flat Spectralon insert supplied or a Spectralon standard (5 in Fig. 101) to be ordered separately can be used as reference.
3. Place the sample in the integration sphere and carry out the sample measurement.

## 24.4 Maintenance and care

Please observe the following:

- In spite of all care it might be necessary to clean the mirrors, in particular mirror 3. Only use dry, clean, i.e. oil-free compressed air or nitrogen for this purpose.
- Clean the Spectralon contact surfaces with the hair brush or with dry, clean compressed air, rinse with distilled water and then blow dry.
  Remove stubborn contamination using ether, ethanol or methanol or fine-grained sanding paper (grain size 220 – 240) and then rinse with distilled water and blow dry with compressed air.
- When cleaning standard samples always observe the manufacturer instructions.

# 25 Base plate with aperture baffle

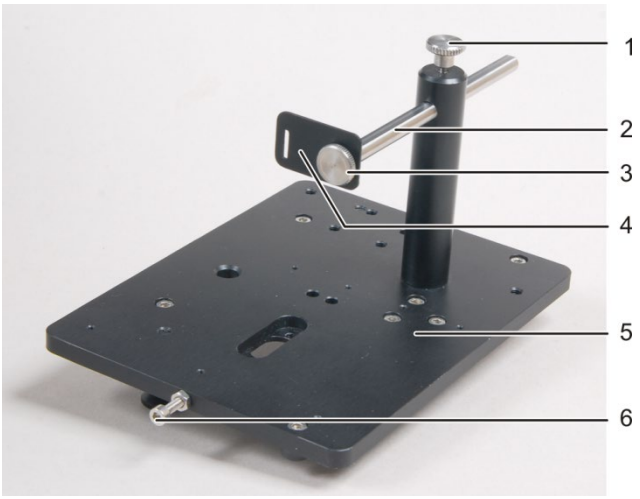
The aperture baffle can be installed in the sample chamber in addition to the cell holders and cell changers. It can be mounted either on a separate base plate or on the base plate for the cell changers. In addition, the separate base plate can be used for individually designed experiments.

The baffles supplied cut the beam down to the defined geometric shapes. They thus make it possible to use microcells or semi-microcells without blackened rims.

The baffles are supplied in the sizes 3 x 3 $mm^2$, 6 x 1 $mm^2$ and 9 x 1.5 $mm^2$. Customer-specific baffles with different dimensions can also be supplied.

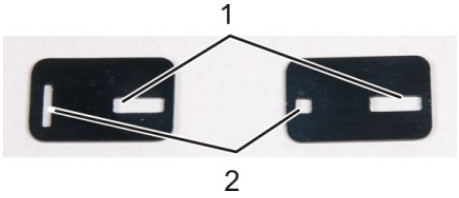
If micro cells without blackened rims are used in the 8-cell changer, it is recommended to insert an additional baffle into the cell duct for cloudy samples (see below). The baffle separates radiation that is reflected at the cell walls multiple times and therefore is at an oblique angle to the optical axis.

Layout




1 Knurled head screw for fastening the baffle mount
2 Baffle mount
3 Knurled head screw for fastening the baffle
4 Baffle
5 Base plate
6 Stop bolt

**Fig. 108 Base plate with baffle**




1 Slots to attach on the baffle mount
2 Baffle apertures

**Fig. 109 Baffles for base plates and baffle for the cell duct for cloudy samples**

Installing the base plate

1. Screw the support rods in the sample chamber into the lower position (→ "Converting the sample chamber" p. 9).
2. Place the base plate onto the support rods and move into the desired position. Press down the base plate until it engages on the support rods with a click.
3. Loosen the knurled head screw (1 in Fig. 108) and slide the baffle as close as possible to the sample. Tighten the knurled head screw.
4. Adjust the baffle:
   - Configure the zero order of the optical beam (→"Configuring the zeroth order" p. 11).
   - Hold a white paper strip as a screen at the later position of the sample.
   - Loosen the knurled head screw (2 in Fig. 108) and slide the baffle until the optical beam hits the sample optimally.

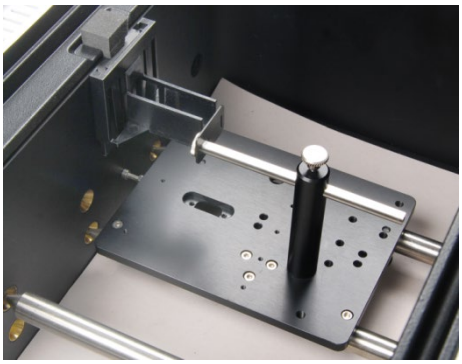


**Fig. 110 Base plate installed in front of the standard cell holder**

Installing the aperture baffle on the cell changer.

The aperture baffle can be installed on the base plate of any 6-cell and 8-cell changer (→ "Using the aperture baffle" p. 36).

# 26 Scanning attachment for solid samples

The scanning attachment for solid samples is used for the locally resolving determination of the transmittance of small transparent solid samples.

As a sample holder the holders for solid samples or a holder with support rods similar to the holder for round cells and alternatively be used. The adapter of the sample holders can be removed and replaced by one's own holders.

The maximum scanning range without sample holder is 70 mm, with sample holder 30 mm.

The scanning attachment for solid samples is automatically detected by the ASpect UV software during device initialization.

Layout

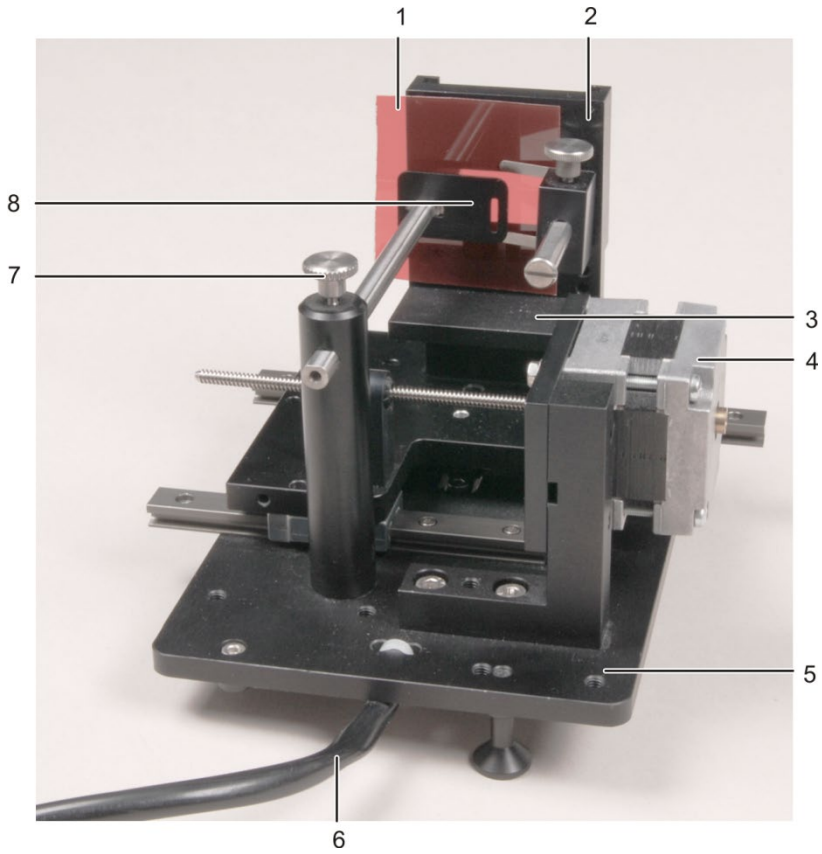



**Fig. 111 Layout of the scanning attachment for solid samples**

1 Sample
2 Holder for solid samples
3 Carriage
4 Step motor
5 Base plate
6 Interface cable to connect to the SPECORD PLUS
7 Knurled head screw for fastening the baffle guide
8 Baffle

Installing the scanning attachment in the SPECORD PLUS and inserting the sample

1. Screw the support rods in the sample chamber into the lower position (→"Converting the sample chamber" p. 9).
2. Place the scanning attachment onto the support rods in the measuring channel.
3. Slide the scanning attachment up to the stop against the front sample chamber wall.

4. Press down the base plate on the right side until the attachment engages with a click.
5. Connect the plug to connector (**M**) in the right sample chamber wall.
6. Secure the samples in the holder (→ "Holder for solid samples" p. 109).
7. Attach the baffle as close as possible to the sample.

   **Note:** The baffle must neither touch the sample nor contact the sample holder when the sample is moved through the beam path.

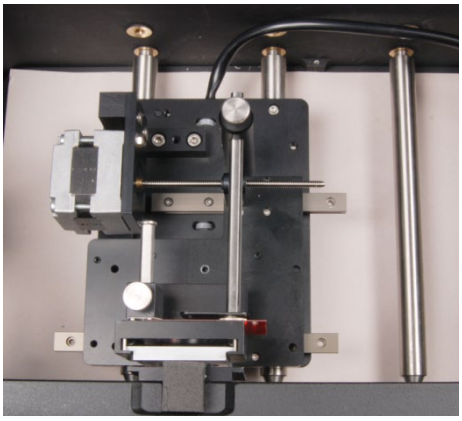


**Fig. 112 Scanning attachment for solid samples installed in the SPECORD PLUS**

Setting the measurement parameters

The scanning attachment is automatically detected during the device initialization in ASpect UV and displayed in the method parameters.

Make the following settings on the ACCESSORY tab:

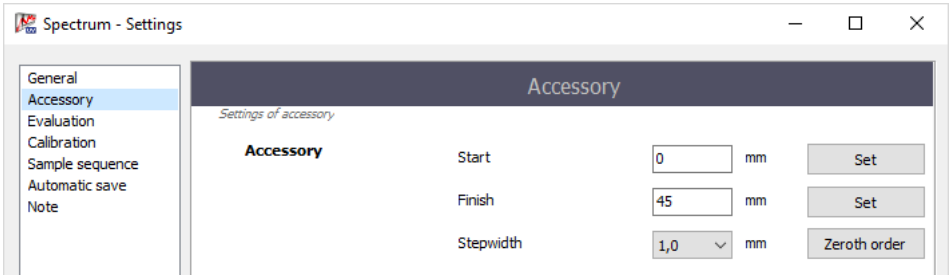



**Fig. 113 Measuring parameter configurations for the scanning attachment**

| Parameter / button | Description |
|---|---|
| START | Start coordinates for a measurement |
| FINISH | End coordinates for a measurement |
| STEP WIDTH | Measuring point spacing on the sample |
| [SET] | Moves the scanning attachment to the set start and end coordinates of the measurement. |
| [ZEROTH ORDER] | Set the monochromator mesh to the zero order.<br>The undispersed (white) light of the Vis lamp passes through the sample chamber and due to its good visibility facilitates the adjustment of the sample in the scanning attachment. |

Positioning the sample

The start and end coordinates are adjusted in the following manner:

1. Use [ZEROTH ORDER] to set the zeroth order of the beam path.

2. In the START field, set a start coordinate for the measurement and click on [SET].

   The scanning attachment moves to the set position.

3. Change the START value until it matches the desired start position on the sample.
4. In the same way, adjust the end coordinate in the END field.

Removing the attachment from the sample chamber

1. Move the scanning attachment into the parking position (INSTRUMENT ▸ ACCESSORY ▸ SAMPLE POSITION / [PARKING] button).
2. In the ASpect UV main window, select INSTRUMENT ▸ ACCESSORY ▸ ACCESSORY OFF.
3. Disconnect the plug from the connector in the right sample chamber wall.
4. Pull up the scanning attachment on the right side of the base plate until it disengages from the clamp on the support rods with a click.
5. Remove the attachment from the sample chamber.

# 27 Fiber coupling with measuring sensors

An adapter is used to couple the optical fibers of measuring sensors to the SPECORD PLUS . The adapter makes it possible to move the measuring position out of the sample chamber. The operating range of the optical fibers is 220 to 1100 nm.

## Attention

**Ageing of optical fibers due to UV radiation!**

Note that the optic fibers may be permanently damaged at wavelengths below 220 nm. Therefore, only work in the spectral range above 220 nm.

Layout of the fiber coupling with measuring sensor

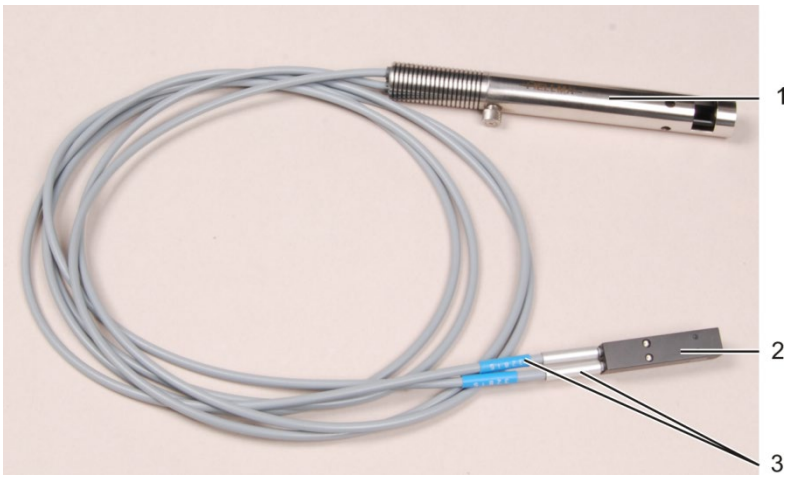
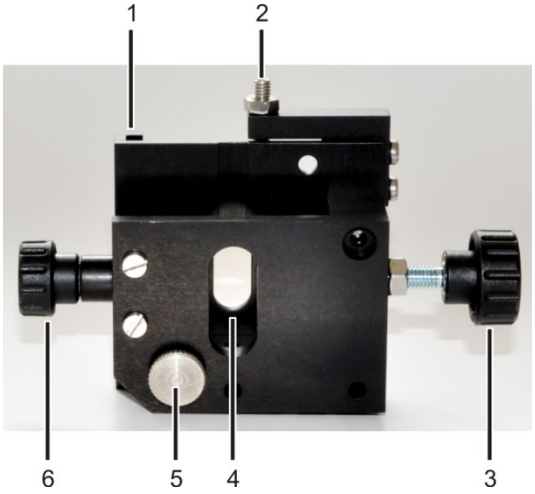



**Fig. 114 Immersion sensor for transmittance**

1 Measuring sensor
2 Optic fiber adapter
3 Optic fiber




1 Guide
2 Vertical adjustment screw
3 Horizontal adjustment knurled head screw
4 Duct for receiving the sensor adapter
5 Knurled head screw for fastening the holder
6 Spring catch

**Fig. 115 Adjustable holder to accept the optic fiber adapter**

The optic fiber coupling consists of an adjustable holder and an adapter decoupling the light from the sample chamber and directing it into a fiber cable. This optic fiber cable channels the light to the measuring sensor.

A second optic fiber cable channels the light weakened by the sample in the measuring sensor back to the adapter. The adapter re-couples the weakened light to the measuring channel.

**Attention**

During the measurement, no extraneous light, not even from the measuring sensor, may get into the sample chamber. You should therefore use the measures for the suppression of extraneous light at the sensor provided by the manufacturer.

Layout of the fiber coupling

The standard immersion sensor and the optic fiber adapter require fibers with collimating connectors. Sensors with SMA connectors are connected, via the coupling pieces supplied, to optic fiber cables that are equipped with SMA connectors on one side and with collimators on the other side for connection to the optic fiber adapter in the sample chamber.

Installing the adapter in the sample chamber

Do not kink or squeeze optic fiber cables!

1. Slightly unscrew the locking screw for fixing at the holder (5 in Fig. 115) to prevent jamming when inserting the holder.
2. Slide the holder onto the adapter for cells.
3. Unscrew the left or right side component of the sample chamber (→ "Converting the sample chamber" p. 9).
4. Place optic fiber adapter into the holder: Pull out the spring catch (6 in Fig. 115) a little and place the cell into the duct.
5. Remove two rubber seals that match the diameters of the optic fiber cables from the apertures in the sample chamber wall. Remove the white plugs.
6. Press the optic fiber cables into the slots of the rubber seals.
7. Insert the rubber seal into the sample chamber aperture with the slot pointing down.

   **Important:** The optic fiber cables must not be kinked or protrude into the beam path.
8. Screw on the side component.

   ✓ The adapter is installed in the sample chamber.

Adjusting the adapter

1. Insert the adapter into the holder.
2. Initially, adjust visually:
   - Configure the zero order of the Vis lamp (→"Configuring the zeroth order" p. 11).
   - Monitor through the gap how the light hits the adapter. Using the knurled head screw (3 in Fig. 115) adjust the adapter in such a way that the light hits the adapter at the center.
3. In the PHOTOMETRY module set a method with the following parameters:

   GENERAL tab:

| Parameter | |
|---|---|
| MEAS. MODE | TRANSMITTANCE |
| WAVELENGTHS [NM] | 500.00 |
| INTEGRATION TIME [S] | 0.1 |
| SLIT [nm]<br>(SPECORD 210/250 PLUS only) | 1 |

SAMPLE SEQUENCE tab:

- One reference measurement in first position
- Further sample rows with the sample type SAMPLE for adjustment

4. Start the measurement with  .
5. Perform the reference measurement.
6. Consecutively modify the vertical adjustment (by rotating the adjustment screw 2 in Fig. 115) and the horizontal adjustment (by rotating the knurled head screw 3 in Fig. 115) and each time measure the transmittance **with the sample chamber cover closed**. Repeat the process until the transmittance value reaches its maximum.

   **Note:** Do not modify the vertical adjustment and the horizontal adjustment at the same time.
7. Hold the knurled head screw (5 in Fig. 115) tight in order to secure the holder relative to the beam path.

   ✓ The adapter has been adjusted.

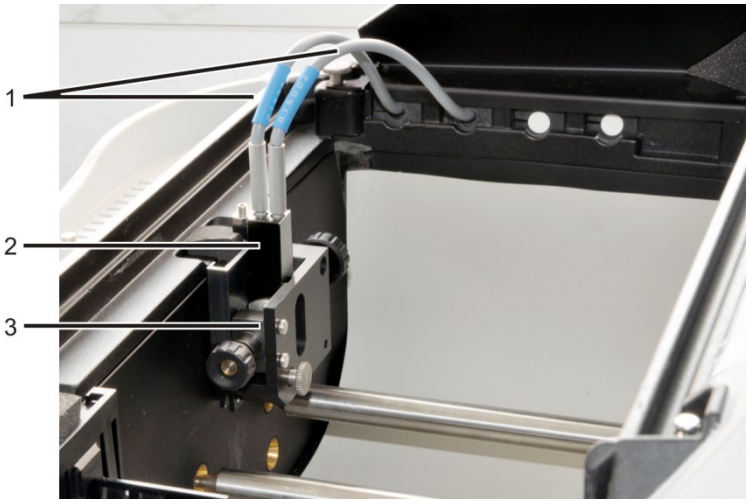



**Fig. 116 Optic fiber adapter inserted in the sample chamber**

1 Optic fiber
2 Optic fiber adapter
3 Adjustable holder