FL 6500/8500 Low Temperature Cell Holder Installation Instructions

This instruction sheet describes the installation of this accessory which is used with the FL 6500/8500 Fluorescence Spectrometer.

NOTE: *Read these instructions before you install this accessory.*

Contacting PerkinElmer

Supplies, replacement parts, and accessories can be ordered directly from PerkinElmer, using the part numbers.

See our website:

http://perkinelmer.com

PerkinElmer's catalog service offers a full selection of high-quality supplies.

To place an order for supplies and many replacement parts, request a free catalog, or ask for information:

If you are located within the U.S., call toll free 1-800-762-4000, 8 a.m. to 8 p.m. EST. Your order will be shipped promptly, usually within 24 hours.

If you are located outside of the U.S., call your local PerkinElmer sales or service office.

Features

- Easy to install
- Easy to use
- Used for measurement of fluorescence and phosphorescence at a liquid nitrogen temperature

Figure 1 Low Temperature Cell Holder (P/N:N4201045)

**PerkinElmer, 710 Bridgeport Avenue,
Shelton, CT 06484-4794, U.S.A**

Produced in the USA.

Dimensions and Specifications

Physical Characteristic	Specifications
Cooling System	Liquid nitrogen
Cooling Temperature	77K (-196℃)
Liquid Nitrogen Reservoir (L)	0.84
LT Cell Holder Dimensions (mm)	197(W) x 275(D) x 177.5(H)
Weight (Kg)	2.48
Accessory Cover Dimensions (mm)	180(W) x 90(D) x 83.5(H)
Cover Weight (Kg)	0.22

Configuration of the Low Temperature Cell Holder

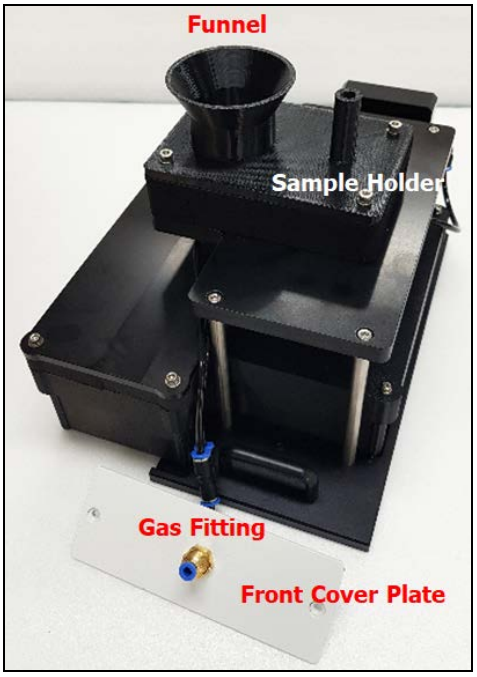


Figure 2 Low Temperature Cell Holder

Figure 3 Accessory Cover

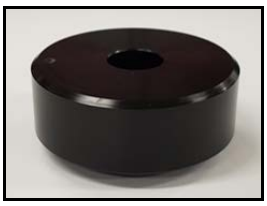


Figure 4 Cover for Alignment

Figure 5 Teflon Rod

NOTE: *NMR Tube for experiment is required to use this accessory. The NMR Tube is not included in the accessory configuration and should be purchased separately. We recommend a quartz material tube of 5mm outer diameter, 3.4mm inner diameter and 7 inches length.*

Installation

1. Prepare the FL 6500/8500 Fluorescence Spectrometer to install this accessory.
2. Connect the instrument power cord and the communication cable.
3. Loosen the accessory fixing bolt and remove the current sample accessory.

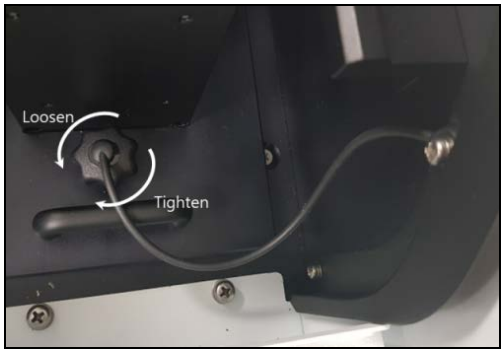


Figure 6 Loosening the accessory fixing bolt

4. Pull out the cell holder by hand.

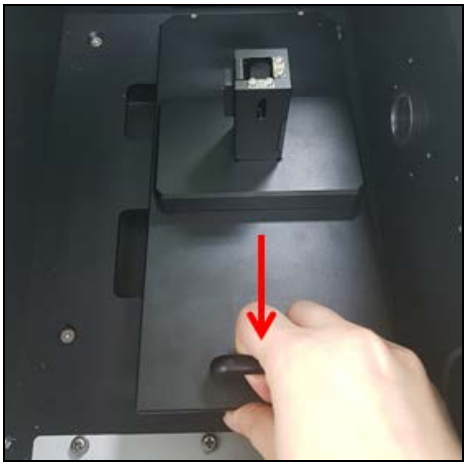


Figure 7 Pulling out the cell holder

5. Remove the current front cover plate.

Figure 8 Front Cover Plate

6. After checking the pogo pin position in the sample compartment, place the Low Temperature Cell Holder to be fit into the pogo pin well.

Figure 9 Installing the accessory

7. With the bolts secure the front cover plate of the Low Temperature Cell Holder.

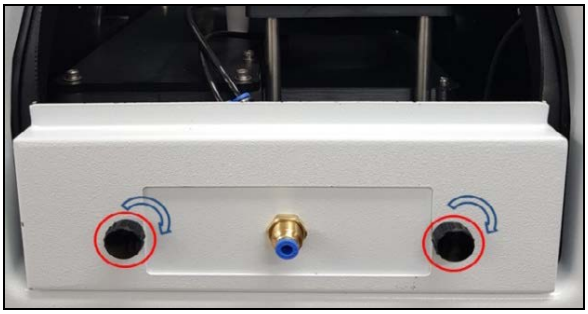


Figure 10 Tightening the bolts

8. Tighten the accessory fixing bolt again.

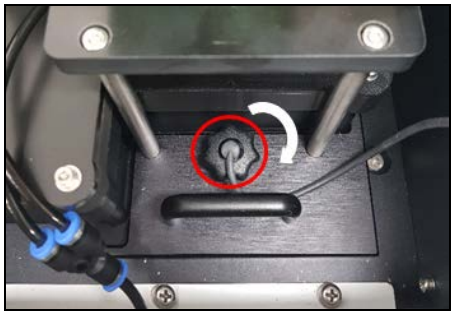


Figure 11 Tightening the accessory fixing bolt

9. With opening the lid door, then close the sample compartment lid, and then mount the accessory cover.

Figure 12 Mounting the accessory cover

10. Connect the gas flow. (Go to page 9 “Purging the instrument.”)

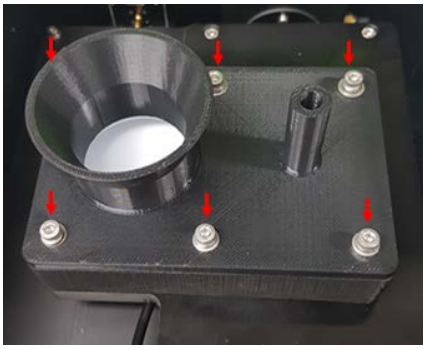
Figure 13 Purge gas flow connection

11. Switch on the instrument.

Alignment

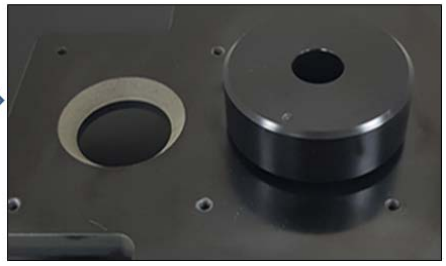
NOTE: *When the Low Temperature Cell Holder is installed for the first time and the unexpected peak is shown, you must align to achieve the optimal spectrum. This must be performed at room temperature.*

1. Loosen the six M4 screws, and then remove the top cover.

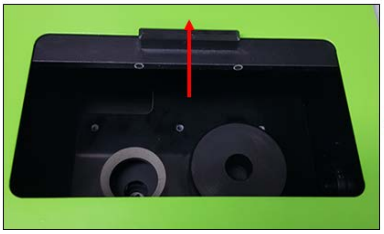


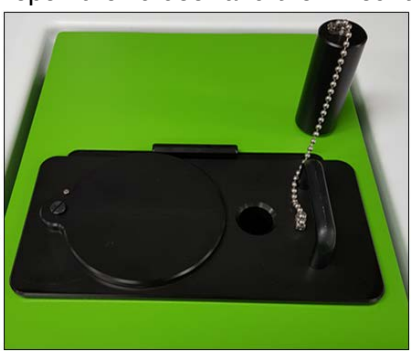
2. Loosen (but do not remove) the four screws (see the red circle in the following photo).

3. Insert the alignment cover into the hole of the screws.



4. Close the sample compartment lid and open the lid door and then mount the accessory cover.

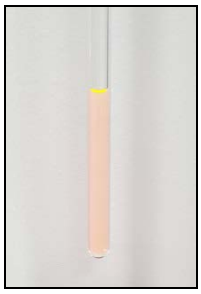




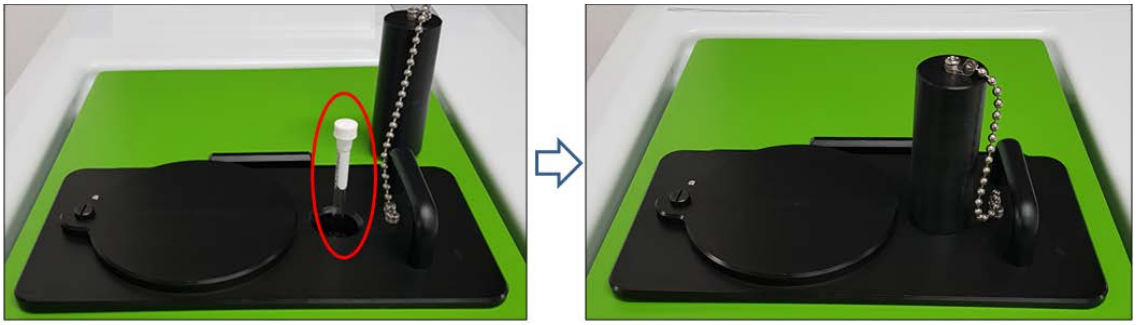
5. Prepare a solution of a suitable fluorescent sample.

   For example, 10 µM Rhodamine 6G in Ethanol.

6. Fill one of the sample tubes to a depth of 35 mm with the sample.



7. Wipe the outside of the tube to dry it, and then insert the tube gently down and close the sample cover.



8. In Spectrum FL, start the Spectra Scan mode and enter the following parameters:

   Scan Type: Emission Scan

   Scan Mode: Single Scan in Intensity

   Luminescence Mode: Fluorescence

   Excitation wavelength: 490 nm

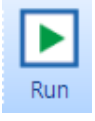
   Emission wavelength: 510 nm ~ 700 nm

   Initial Dark: True

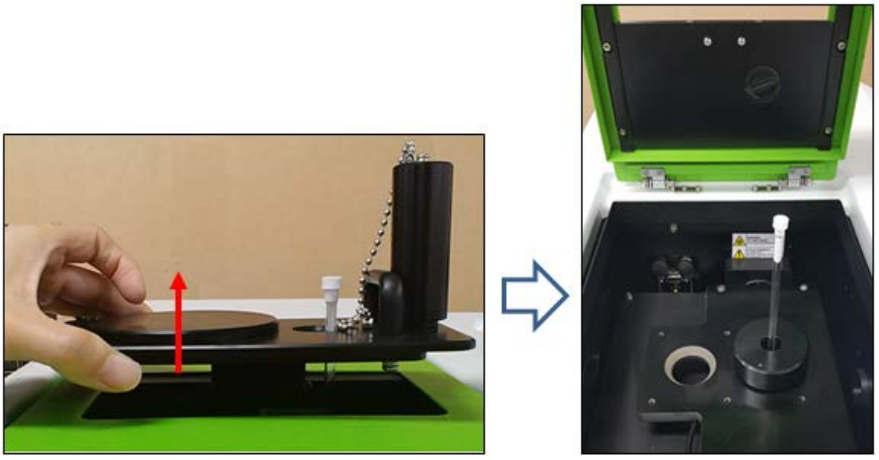
   Excitation slit/Emission slit: 10 nm / 10 nm

NOTE: *These parameters are only for using Rhodamine 6G as the sample. If another sample is used, then the excitation and emission wavelength will have to be set accordingly.*

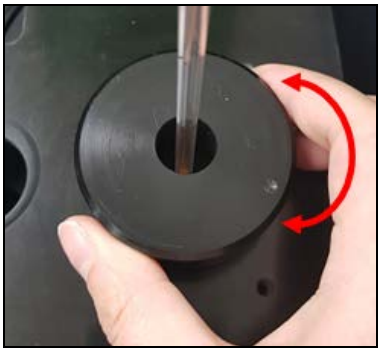
9. Save the method, and then click **Run** icon.



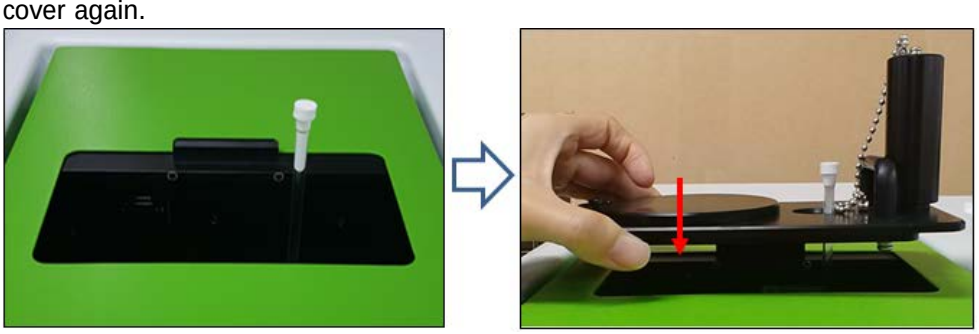
10. When the measurement is finished, check if the spectrum shape looks similar with the optimal spectrum of #15. If you've got the good spectrum, you can skip steps 11 to14.
11. Remove the accessory cover and open the sample compartment lid carefully.



12. Turn the alignment cover slightly to change the angle of the inner copper block.

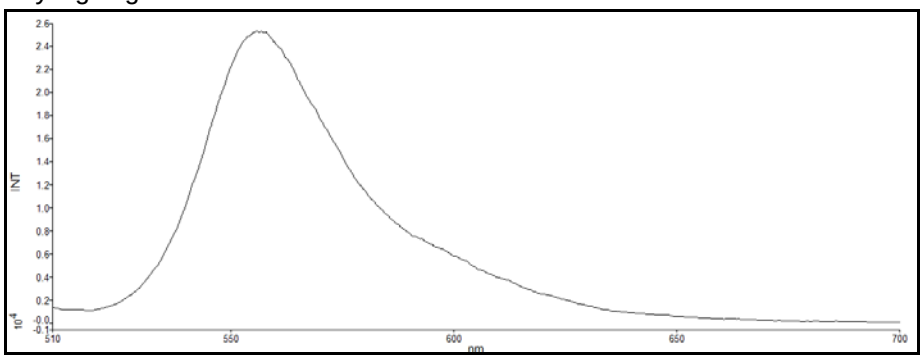


13. After opening the lid door, close the sample compartment lid, and then mount the accessory cover again.



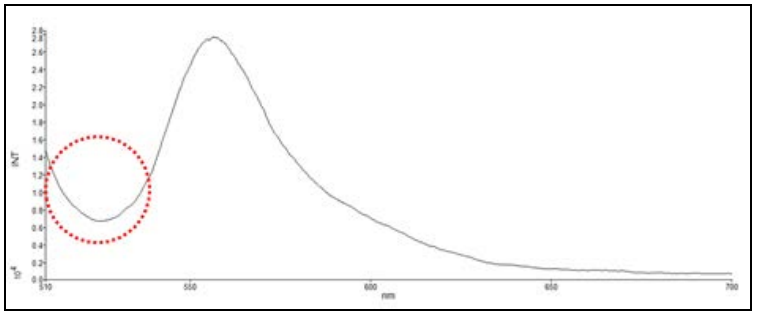
14. Measure the sample again. Repeat the procedure until a good Spectrum is obtained.

15. The following spectrum shows the optimal angle of separation between fluorescence and Rayleigh light.



NOTE: *If fluorescence and Rayleigh are overlapping, the angle needs to be adjusted. (see step 12)*

Examples of erroneously measured spectra



16. After achieving the optimal angle, remove the sample tube and the alignment cover.
17. Tighten the four securing screws.

CAUTION	*While tightening the screws, be careful since the position of the sample tube can be shifted.*
ATTENTION	*Lors du serrage des vis, faites attention car la position du tube à échantillon peut être décalée.*

18. Place the top cover of the accessory, and secure it using the four screws removed previously.

Purging the instrument

Before measurement of the liquid nitrogen temperatures can be performed, the instrument must be purged with dry gas, for example helium or nitrogen. This gas must be free of carbon dioxide and moisture.

The purpose of purging is to avoid the build-up of ice on optical surfaces inside the instrument, in the sample area and on the sample tube itself. A gas pressure of 0.025 MPa is normally sufficient.

The gas flow must be connected to the one touch fitting on the front cover plate as shown below. Tubing O.D size is 4 Ø.

Filling the accessory with liquid nitrogen

WARNING

During filling of the liquid nitrogen tank, protective gloves must be worn, and care must be taken not to touch the metal surfaces of the accessory with unprotected hands.

AVERTISSEMENT

Lors du remplissage du réservoir d'azote liquide, il faut porter des gants de protection et veiller à ne pas toucher les surfaces métalliques de l'accessoire avec des mains non protégées.

WARNING

Due to the danger of asphyxiation please make sure that the area in which the instrument/accessory and liquid nitrogen vessel are located is well ventilated.

AVERTISSEMENT

En raison du risque d'asphyxie, veuillez vous assurer que la zone dans laquelle se trouvent l'instrument/l'accessoire et le récipient d'azote liquide est bien ventilée.

1. During filling, an enclosed Teflon rod should be located in the accessory to avoid icing in the sample holder position. Gently push a Teflon rod into the sample holder.

NOTE: *Some resistance will be encountered when the Teflon rod comes into contact with the two clips which secure the teflon rod. Turn the teflon rod if necessary to overcome this resistance.*

2. Flow the dry gas through the purge connection.
3. Open the funnel cover and carefully pour liquid nitrogen into the funnel. This will require approximately one liter of liquid nitrogen. Pour slowly until the liquid nitrogen appears and the tank overflows. When the copper finger gas almost reached temperature, the liquid nitrogen will boil for several seconds, after this the system settles down.

4. When the boiling has subsided, the tank should be slowly filled to overflowing.
5. Close the funnel cover, remove the Teflon rod and insert the sample tube.

NOTE: *To maintain constant temperature, approximately one liter of liquid nitrogen will be required for each hour of use (depending on room temperature). The accessory should be topped up with liquid nitrogen every 10 to 15 minutes to ensure temperature stability.*

Measurement

1. Open the **Spectrum FL** software and select a measurement mode.
2. Check the recognition of the accessory.

3. Set up the measurement parameters.
4. Click **Save** to save the method after setting up the parameters.
5. Prepare a solution of the sample for measurement.

   Suitable solvents are ethanol, cyclohexane or methylcyclohexane or a mixture of diethylether, isopentane and ethanol (EPA) in the ratio 5:5:2.

CAUTION: *Do not use water as a solvent, the pressure caused by freezing will burst the capillary.*

6. Using a narrow pipette, fill a clean, dry sample tube to a depth of at least 35 mm (equivalent to approximately 320 µL of sample). Ensure that the sample is free of bubbles.
7. Wipe the outer surface of the tube dry.
8. Remove the Teflon rod from the sample holder and replace with the sample tube, close the sample cover.
   The sample cover should be open for as short a time as possible to avoid condensation build-up in the sample holder area.

9. The sample should require about two to three minutes to reach 77 K.
10. Select the **Run** icon.
11. Input the Experiment name and select **OK**.
12. Measure the sample fluorescence or phosphorescence as desired. If the measurement takes more than ten minutes, the sample tube should be rotated through 360 degrees to avoid liquid carbon dioxide building up in the sample holder area.

NOTE: *A potential problem is snowing of the solvent. In this case, the solvent does not freeze to a clear glass, but shows white flecks. This can be determined by rapidly removing the tube and observing it. If snowing occurs, this does not prohibit qualitative and quantitative analyses, but gives much higher background levels and lowered sensitivity. The snowing effect may be removed by changing the solvent. Methylcyclohexane, for example, forms a clear glass, while hexane forms snow.*

NOTE: *The circular cross-section of the sample tube can cause stray light to be reflected into the emission light path. Select one of the emission cut-off filters to remove second and third order stray light from the spectra.*

13. When the accessory returns to room temperature after use, it can accumulate condensation from water. If the accessory is to be used again within several hours, ensure that the copper finger is dry by wiping with a paper tissue.

NOTE: *While the accessory is at liquid nitrogen temperature, the sample position should never be empty: if necessary, insert a Teflon rod into the tube holder to avoid this. The empty sample position can accumulate CO_2 ice, making it impossible to insert a sample. To prevent this from happening allow the accessory to return to room temperature, insert a Teflon rod into the tube holder and repeat the cooling-down process.*

Removing the Accessory

WARNING

Always allow the accessory to return to room temperature before removal and handling.

AVERTISSEMENT

Laissez toujours l'accessoire revenir à la température ambiante avant de le retirer et de le manipuler.

1. Turn off the purge supply to conserve gas
2. Loosen the accessory fixing bolt and separate the accessory.
3. Store the accessory in a safe place.