Guidelines for the Installation and Setup of the W-Band Light Fiber Sample Rod E600-1021RL

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Scope of Delivery
The following parts are delivered with Bruker's E600-1021RL light fiber sample rod:

(i) Light fiber sample rod with F-SMA connection.
(ii) Sample cramp for 0.9 mm o.d. sample tubes (already attached to the sample rod)
(iii) These installation instructions

Functional Parts of the Light Fiber Sample Rod
In opposite to the standard W-band sample rod, this light fiber sample rod has some additional features.

- The length of the sample rod between the sample cramp and the stopping ring has been adjusted to fit to the standard length of the W-band resonator. Careful optimization is required though (see below)
- In case of a precooled cryostat, insertion of the sample rod takes at risk to introduce air and moisture into the cavity and/or cryostat. To this end, an O-ring closure, located at the top part of the sample rod, can be pushed upwards.
- At the top an F-SMA male connector is attached for interconnection of the light fiber within the sample rod to an external light fiber.
- The distance between the lower end of the light fiber and the sample tube can be adjusted (cf. Fig. 1). Height adjustment proceeds via the two screw-nuts. Note, the tread of the screw does not prolong into the support. It is only held in place with O-rings.

Figure 1  Top part of the sample rod
- The sample rod is equipped with a so-called irradiation shield at the bottom part. This shield will protect already frozen samples within the tube from too rapid thawing. It can be shifted upwards to get access to the sample cramp (cf. Fig. 2). Within the resonator it will also be shifted away and will not interfere with the lower part of the resonator.
Installation Procedure: Height Adjustment of the Sample Rod

This procedure will optimize the length of the sample rod. This way, the sample cramp will be in the right position with respect to the plunger of the W-band cavity.

1. Switch on your E600/E680 W-band ELEXSYS system, connect to the spectrometer and set the microwave bridge into tune mode.

2. Adjust the cavity height (empty cavity) such that the tune mode will display the resonance dip of the cavity (the frequency of the W-band bridge should be centered with the frequency slider).

3. On the sample rod, while holding the lower screw-nut rotate the Adjustment Screw counter-clockwise to the upper bound (this will ensure that the light fiber is also at its upper bound).

4. The sample height adjustment on the top part of the W-band probe head should be at zero position.

5. Insert the same rod without a sample tube carefully and smoothly to full length. If a resistance is recognized even if the stopping ring is not on top of the sample height support, do not push further! The sample cramp is already at the lower most position right in its support in the plunger. In case of this, proceed as follows:

   5.1) Lift the sample rod by 1 mm

   5.2) Unfasten the stopping ring and adjust it to fit to the sample height positioning support. Fasten the stopping ring again.

6. In case the stopping ring is touching the sample height support:

   6.1) Unfasten the stopping ring.

   6.2) Insert further the sample rod smoothly and carefully till a resistance is recognized.

   6.3) Lift the sample rod by 1 mm.

   6.4) Adjust the stopping ring to fit to the sample height positioning support. Fasten the stopping ring again.

7. Remove the sample rod completely.

8. Insert again without a sample tube. Check that now the stopping ring is restricting further insertion.

Operation

(1) (1)-(4) of the above Installation Procedure.
(2) Mount a 0.9 mm o.d. sample tube to the sample cramp. The distance of the end of the cramp to the end of the sample tube should be about 18 mm.

(3) Insert the same rod carefully and smoothly; watch the resonance dip of the W-band cavity in the tune mode.

(4) If the dip starts to shift in frequency (downward shift), adjust the frequency tuning on the W-band probe head accordingly.

(5) Follow the dip while further lowering the sample rod.

(6) At one position, the sample tube will reach the bottom plate of the W-band cavity. Further insertion of the sample rod will not shift the frequency of the cavity further. Instead the sample tube will be shift upwards into the cramp.

(7) Further lower the sample rod to full length.

(8) Increase the sample height by 0.5 mm. Now, the cavity dip should shift back in frequency (upward shift in frequency). At this position the end of the sample tube is released from the bottom plate. Rotate the sample rod to optimize the $Q$-value of the cavity.

(9) On the sample rod, while holding the lower screw-nut rotate smoothly the Adjustment Screw clockwise. This will decrease the distance between the lower end of the fiber and the upper end of the sample tube. At one position, the cavity dip will again move downwards in frequency. At this position, the fiber is in close contact to the sample tube.

**Characteristics of the Light Fiber**

The fiber utilized is has the following dimensions:

- **core diameter:** (940 ± 15) µm
- **total diameter:** (1400 ± 50) µm

The pure silica core is surrounded by a cladding, a hard core and finally by a buffer (made out of Tefzel®).

The typical spectral characteristic of the fiber is as follows:

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<th>$\lambda$ / nm</th>
<th>Fiber Transmission in dB/m</th>
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<tr>
<td>248</td>
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