1.7 mm Micro CryoProbe

User Manual

Version 001

Innovation with Integrity

NMR

This manual was written by

Bruker CryoProbe Department

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1 Information on Handling a 1.7 mm Micro CryoProbe

The handling of the 1.7 mm Micro CryoProbe does not significantly differ from handling of other CryoProbes. CryoProbes are high-tech instruments; they have to be handled with care. Never carry the probe holding the tube or the CryoConnector. Avoid any shocks to the probe. Please refer to the “Warnings and Limitations” sheet delivered with your probe.

Nonetheless there are some differences between the 1.7 mm and larger diameter CryoProbes, because of smaller sample diameter and different probe construction:

- The 1.7 mm Micro CryoProbe has a big advantage compared to all other Bruker NMR probes: tuning and matching are very robust to sample changes. You can adjust them once and leave them in position even when changing between samples with different solvents and different salt concentrations. The slight detuning or mismatch that might be observed does not significantly affect the probe performance. Even when using lossy samples it is generally sufficient to adjust the pulse width (p1) without tuning and matching. Only for extreme salt concentrations beyond 500 mMol matching might be necessary.

  Eventually after a re-cool down tuning and matching might have to be readjusted. When you tune or match the ^{1}H channel you should also check tuning of the X- nuclei. Furthermore you should always shim on-axis after ^{1}H tuning and matching.
- Never move the probe inside the magnetic field while it is cold. This might deteriorate the shim quality and make shimming of the probe impossible. If shimming of the probe is seriously deteriorated, thermal cycling of the probe might fix this problem (warm-up/cool-down).
- **Proton pulses require extremely low power. To avoid incidents to the probe the power check option must always be on! In order to work properly the probe must be declared to the spectrometer by running the edhead or edprobe command!**
- **Prosol** should be set correctly once the pulse angles are determined (type **edprosol** in the command line).
- Always work with **rpar** and **getprosol**, never copy data/parameter sets from a RT probe or a 5 mm CryoProbe without changing the power levels for proton pulses.
- Every sample should be cleaned with alcohol and lint free paper or tissue, because the probe is very sensitive. The samples must be free of particles to avoid pollution of the probe. Never use other than original Bruker sample tubes.

Information on Handling a 1.7 mm Micro CryoProbe

2 Typical Pulse Widths and Power Levels

The proton coils in the 1.7 mm Micro CryoProbe are very small and highly efficient; therefore the pulse power levels needed for a 90° pulse are extremely low. Typical power levels used with conventional probes and 5 mm CryoProbes will destroy your Micro CryoProbe. Hence you should always ensure that the power check option is on and the correct probe is declared to the spectrometer.

Typical values for standard instruments are listed as follows:

- The ^{1}H 90° pulse might be found at 10 µs using a power level of about 0.3-0.5 W.
- The ^{13}C 90° pulse might be found at 12 µs using a power level of about 60-120 W.
- The ^{15}N 90° pulse might be found at 25 µs using a power level of about 120-230 W.

These values might vary substantially from one instrument to another and the 90° pulse widths and power levels have to be calibrated on each instrument individually. Always refer to the "Limitations and Warnings" and the "Customer Certificate" delivered with your probe! Never use power exceeding the limits defined by these documents! The power output of your instrument was determined with the amplifiers, boards, cables, and routing present at the time of the measurement. Any changes on your instruments hardware might affect the power output. The best way to protect your probe is to have your instrument re-cortabed following any changes in hardware or routing.

Typical Pulse Widths and Power Levels

3 Information on 1.7 mm Sample Handling

The 1.7 mm sample tubes are very small but still quite robust. Normally, they do not break if they are dropped.

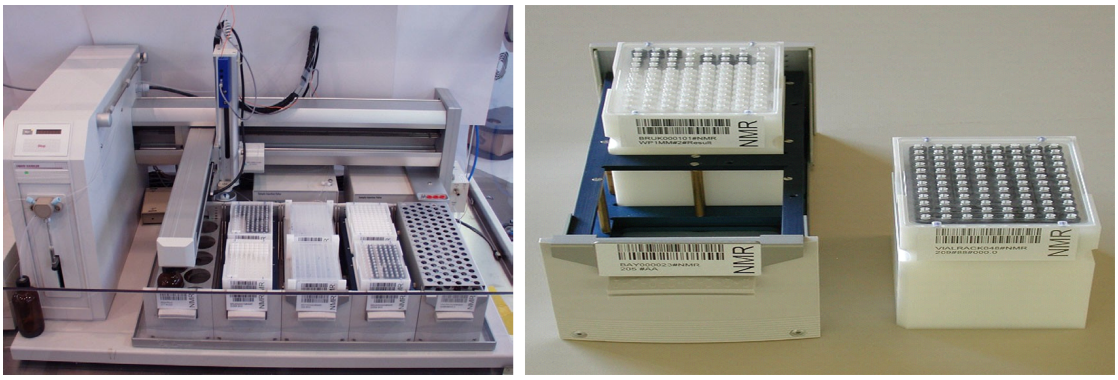

Figure 3.1: Automatic Filling of 1.7 mm Samples with a Gilson 215 Liquid Handler

3.1 Automatic Sample Filling

The most efficient way to fill 1.7 mm sample tubes accurately and fast is to use a sample filling robot, *e.g.* the Gilson 215 liquid handler with the 1 – 1.7 mm accessory which is distributed by Bruker BioSpin (see figure above). However, here we describe some ways of manual sample filling which proved to work well for small sample quantities.

3.2 Manual Sample Filling using Syringes

To fill the tubes use a small (1 ml) syringe and a disposable needle with 0.6 mm outer diameter. The 1.7 mm NMR tubes include an attached capillary holder including a small funnel that helps to guide the needle into the 1.7 mm glass tube. In general the needle will be too short to go all the way down to the bottom of the glass. Fill the tube with liquid to a total height of at least 23 mm. If the needle is too short, such that this is not possible, fill less and repeat several times until the total filling height is reached. In order to bring all the liquid to the bottom, shake or centrifuge the sample tube. After the sample is filled, the tubes can be closed with a plastic ball, which is pressed into the cavity of the funnel. You can either use the pinch tool or just place the plastic ball on a table and press the capillary holder upside down on the plastic ball. Your sample will remain in the bottom of the tube due to capillary forces even if the tube is returned. Please note: the plastic ball does not permanently seal the samples. Its function corresponds to the plastic caps for 5 mm tubes and is only intended for temporary closing.

Another manual possibility to transfer a limited sample quantity is to use a micro-liter HPLC (Hamilton) syringe with a needle of 0.7 mm outer diameter or less.

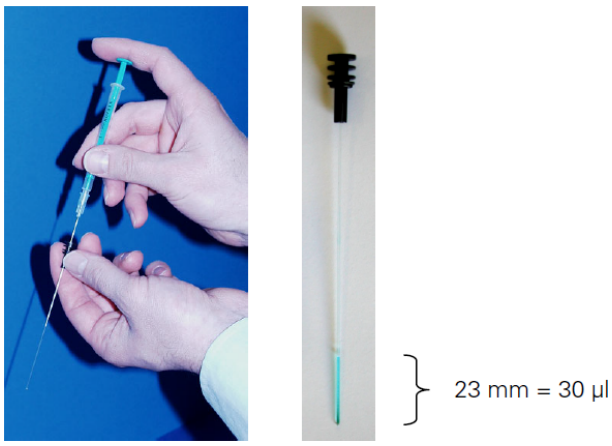

Figure 3.2: 1.7 mm Sample Filling with a Pipette and Centrifuge, or with a Syringe

Sometimes it might be preferable to fill 1 mm sample tubes instead of 1.7 mm tubes for very limited sample quantities. The probe is not specified to work with those smaller diameter samples as the sample is not perfectly held in place but no damage to the probe will occur. There might be problems with sample vibrations or temperature gradients as well as shimming problems since 1 mm samples are not entirely centered in the 1.7 mm probe.

3.3 Sample to Magnet Handling

Once the sample is filled, the tubes are dropped into a shuttle and then can be handled like any other NMR sample. No adjustments concerning sample height and centering are necessary. There are two types of shuttles. One is intended for manual use and allows the usage of automation devices such as the BACS sample changer, the other is intended for use with the SampleJet robot system. The part numbers can be found in the last section.

The Micro CryoProbe is compatible with the high throughput concept and can be used with SampleJet or BACS sample changers. For 1 mm and 1.7 mm samples the sample position and filling height is different from 3 mm and 5 mm samples.

Never use the Bruker Match accessory with a Bruker MicroProbe or a Micro CryoProbe! This will damage your probe and the samples. The Match accessory is intended for measuring small diameter samples in 5 mm probes in a manual fashion.

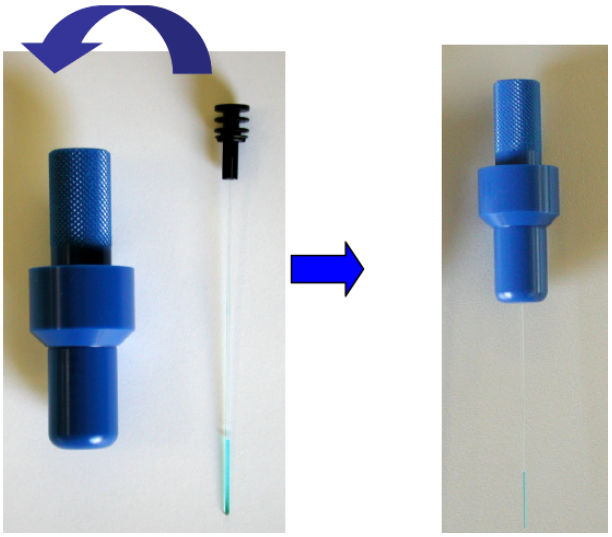

Figure 3.3: Sample and Shuttle Handling

4 Working with 1.7 mm Samples in a Micro CryoProbe

Once the samples are in the shuttle there is no difference between using 1.7 mm NMR tubes in a dedicated 1.7 mm Micro CryoProbe and 5 mm NMR tubes in a 5 mm probe. Please use only Bruker 1.7 mm tubes and shuttles with your 1.7 mm probe (RT or CryoProbe). The tolerances on sample diameter are very tight and the use of different samples might result in damage to the probe. The filling height has to be at least 23 mm (corresponding to 30 µl) in order to achieve good shim results. The samples are to be used non-spinning. If you turn the spinning air on, probe and sample will be harmed because only the shuttle will spin. Nonetheless, in order to avoid vibrations you should have the spinning air turned off. When the sample is inserted in the magnet using a shuttle without the black and white reflective strip, the sample missing LED on the BSMS panel will remain on, even if the sample is down.

You should always clean samples with ethanol and lint free paper before inserting the sample into the probe. **The Micro CryoProbe is very sensitive and can detect the smallest amount of grease on the sample. Furthermore once lint, dust and particles are inserted into the probe they are very hard to remove and the probe might have to be shipped back to Bruker for cleaning or replacement of the center tube.**

Working with 1.7 mm Samples in a Micro CryoProbe

5 Sample Temperature Control

Because of the small sample diameter and the corresponding smaller central tube, the recommended VT-gas flow is lower than for standard 5 mm probes. Maximum and recommended flows for your probe can be found with edhead/edprobe under **Manage** | **View Properties** within the Temperature Controlling Gas Flow parameters. If the VT-air flow is too high the sample might not insert correctly. If you have problems with a temperature gradient in your sample you can increase the airflow once the sample is in place. You can adjust the air flow by typing **edte** in XWIN-NMR or TopSpin. You should calibrate the sample temperature and enter the corrections in **edte**.

Please use the 99.8% Methanol-d4 sample for temperature calibration. The part numbers can be found in *Accessories and Consumables [▶ 17]*.

Calibration of the temperature can be performed using the **calctemp** au program. The sample temperature range is limited to 0 °C to +80 °C. You should never exceed this temperature range, since damage might occur to your probe!

Sample Temperature Control

6 Shimming a Micro CryoProbe

6.1 Manual Shimming

With a Micro CryoProbe shims have less influence than experienced with a 5 mm probe, because the sample diameter is smaller and the coil window is shorter. Also the higher order shims are rather ineffective and therefore do not need to be used. This means that shimming is much simpler compared to a 5 mm probe. In general, in order to shim a probe only the following shims are of importance (shims in square brackets may eventually also be useful). All others should be set to zero, to reduce the overall shim currents:

Z, Z2, Z3, Z4, Z5, [Z6], X, XZ, XZ2, XZ3, Y, YZ, YZ2, YZ3, XY, XYZ, [XYZ2] X2-Y2, [(X2-Y2)Z], [X3], and [Y3].

Upon sample changes only Z, Z2, Z3 and eventually Z4 have to be readjusted. The shim settings are also dependent on the sample filling height and the solvent used. If the filling height and solvent do not vary from sample to sample, often there might be no need to shim when changing between two samples (e.g. when the 1.7 mm samples were automatically filled using an automatic liquid handler). Any time the ^{1}H channel is tuned and/or matched shimming might be necessary on low order on-axis shims.

6.2 Autoshim

Since there are only a few shims used for the 1.7 mm MicroProbe, **autoshim** is an effective, simple and convenient option. This is an example for tune file for auto- shim. It can be created in XWIN-NMR or TopSpin by the command **edtune**. This file should be sufficient to shim after sample changes:

```
AUTOSHIM Z=2 Z2=5 Z3=10 Z4=25
AUTOSHIM X=5 Y=5
```

You will have to save this tune-file. Autoshim can then be started with these parameters by the command: **tune TUNEFILENAME** from the command line in XWIN-NMR or TopSpin.

6.3 Gradient Shimming

The new gradient shimming program “Topshim” can be used for automatic shimming. It is available since TopSpin version 2.0 on all Avance consoles with SGU. Please refer to the manual e.g. by typing **topspin help** in the command line. We suggest using the command **topshim** for on-axis shimming (eventually with the option **topshim z6**, if necessary) and **topshim 3d ordmax=5,4** for off-axis shimming.

In some rare cases Topshim does not yield optimum results. In this case it might be useful to define the range to be used by Topshim for shimming. This can be done either by typing **topshim zrange=0.6** to limit the shimming range to +/- 0.6 cm or by adding the parameter **zrange=0.6** in the **topshim gui** window.

Alternatively the old gradient shim program “gradshim” can still be used. If you have the required hardware this shim program can be set up during the installation of your probe by a Bruker service engineer. There is no difference in using 1D gradient shimming with 1H or 2H or 3D RCB gradient shimming on a 1.7 mm Micro CryoProbe and on 5 mm CryoProbes. The only difference is that you should avoid shimming high off-axis and on-axis shims (see list above in *Manual Shimming [▶ 15]*) since they are highly inefficient on the small sample diameter and will heavily increase the total shim currents without adding any extra performance.

Shimming a Micro CryoProbe

The parameter files for gradient shimming of a 1.7 mm CryoProbe are different from the parameter files of a 5 mm CryoProbe. If the probe is correctly declared to the spectrometer using the **edhead** or **edprobe** command then the correct parameter set is always automatically chosen. When using the wrong parameters no damage will occur to your probe, but the shim results will not be satisfactory.

7 Accessories and Consumables

Part Number	Quantity	Description
Z106462	1	Set of 96 1.7 mm tubes in a rack (compatible with Gilson and SampleJet). 1.7 mm tubes with data matrix (SampleJet compatible cap with hole attached), bulk discount.
Z107504	1	Set of 96 1 mm tubes in a rack (compatible with Gilson and SampleJet). 1 mm tubes with data matrix (SampleJet compatible cap with hole attached), bulk discount.
Z72497	1	Set of 100 polished 2.5 mm balls (POM), white. Balls used to close the 1 mm and 1.7 mm tubes permanently.
Z147552	1	Set of 100 polished 2.5 mm balls (POM), pastel orange. Balls used to close the 1 mm and 1.7 mm tubes permanently.
Z147553	1	Set of 100 polished 2.5 mm balls (POM), azure. Balls used to close the 1 mm and 1.7 mm tubes permanently.
Z147554	1	Set of 100 polished 2.5 mm balls (POM), traffic red. Balls used to close the 1 mm and 1.7 mm tubes permanently.
Z147555	1	Set of 100 polished 2.5 mm balls (POM), zinc yellow. Balls used to close the 1 mm and 1.7 mm tubes permanently.
Z72498	1	Pinch tool. Tool to press the balls into the 1.7 mm funnel cavity – use for tubes in racks.
Z106492	1	1 mm and 1.7 mm plastic shuttle for manual use or BACS (works for both 1 mm and 1.7 mm tubes). Like a spinner for 5 mm tubes, usable with BACS, bulk discount.
Z106558	1	1.7 mm plastic shuttle for SampleJet robot. Enables the SampleJet robot to work with 1.7 mm tubes (one shuttle per SampleJet).
Z107648	1	1 mm plastic shuttle for SampleJet robot. Enables the SampleJet robot to work with 1 mm tubes (one shuttle per SampleJet).

Table 7.1: 1.7 mm Accessories and Consumables

Part Number	Quantity	Description
Z10717	1	Lineshape test sample 1 % CHCl3.
Z10718	1	Sensitivity test sample 0.1 % Ethylbenzene (EB).
Z10719	1	Water suppression 2 mM Sucrose in 10 % D2O / 90 % H_2O.
Z10721	1	15N/13C pulse calibration test sample 100 mM Urea.
Z10724	1	13C sensitivity and lineshape test sample ASTM.
Z10727	1	Calibration sample “Doped Water” 0.1 mg/ml GdCl3 in D_2O.
Z10734	1	Low temperature calibration sample 99.8% Methanol-d4.

Table 7.2: 1.7 mm Standard Test Samples

Part Number	Description
AH0340VAR17	Complete Gilson system to fill 1.7 mm tubes in 96 well plate type racks, including 2 sets of 96 1.7 mm tubes (2 x Z106462). The robot can be set up to fill also 1 mm, 3 mm or 5 mm tubes with no additional parts required. A Windows PC to control the robot is required and is not part of the shipping.
AH0341VAR17	UPGRADE for an existing Gilson Robot to fill 1.7 mm tubes in 96 well plate type racks, including 2 sets of 96 1.7 mm tubes (2 x Z106462). The robot can be set up to fill also 1 mm, 3 mm or 5 mm tubes with no additional parts required. A Windows PC to control the robot is required and is not part of the shipping.

Table 7.3: Gilson Filling Robot or Gilson Upgrades

Part Number	Quantity	Description
Z71495	1	1 mm needle for Gilson 215 (replacement).
Z72483	1	Tubing, fittings and bottles for Gilson. Contains spares of tubing, fittings, ferrules etc.
W3003936	1	Barcode label support. For racks used on a Gilson system. One holder per rack.
Z71611	1	B-205 precision Gilson rack. Precision Gilson rack for two 96 tube racks (e.g. Z106462 or Z107504).

Table 7.4: Spare Parts and Other Accessories for the Gilson Filling Robot

8 History

Index	Date	Changes	Initials
00	30.06.2009	New document Number ZTSC0001 / Review (replaced ZTPE0010)	KTI/FRN
01	18.02.2016	Changes: Manual created under part number Z33127 revision 00.	PRST/ SEY

Table 8.1: History

History

9 Contact

NMR Hotlines

Bruker Corporation provides dedicated hotlines and service centers. Please select the NMR service center or hotline you wish to contact from our list available at:

https://www.bruker.com/service/information-communication/helpdesk/magnetic-resonance.html

Contact our NMR service centers, so that our specialists can respond as quickly as possible to all your service requests, application questions, software or technical needs.

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