NMR Spectroscopy Hardware Guide

In the context of uniaxial strain measurements

Version 1.0.0

D. V. Garrad

August 26, 2025

Contents

1 Introduction

This document is meant to outline the NMR spectrometer setup[1] at LIN[2], Paul Scherrer Institute, Villigen, CH. It is meant as both a reference for experienced users, as well as a starting point for new users.

[1] https://www.psi.ch/en/lin/nmr-spectroscopy
[2] https://www.psi.ch/en/lin

2 Hardware Setup

2.1 The Stick

The "stick" is the device which holds the sample, thermometer, strain cell, and, in general, other auxialiary devices. See Fig. **??**. This device is to be inserted into a PPMS (Fig. **??**) and will transfer the RF signal to the sample. Setting up the stick can be separated into TODO: how many parts.

2.1.1 Mounting the Strain Cell (or PLA stand-in)

The stick that Jonas Phillipe developed was designed for use with a strain cell - specifically, the Razorbill FC1X0[3]. This means that even if you're not performing a uniaxial strain/stress experiment, you'll need to fill the empty space with a 3D printed stand-in (Fig. **??**).

Installing the strain cell is relatively simple - first, thread the coaxial cables for the strain cell and auxiliary devices, as well as the two wires for the RF signal through the bottom of the cell (see Fig. **??**). Then, some brass screws on a bracket (Fig. [**?**]) are screwed into the bottom of the strain cell, and the ones on the wings of the bracket are screwed into the end of the stick itself.

2.1.2 Mounting the Sample

Assuming the sample is nicely shaped and sized (millimeter by millimeter is the best scale for use in a strain cell, TODO: otherwise?), one can prepare Stycast [**?**] or some other glue (e.g., a 100:4 ratio of Stycast to catalyst 9 [**?**] was used in the first uniaxial LBCO experiment). Note that the term "glue" is used somewhat liberally, here, to give a name to whatever is keeping a sample in place. The strain cell has two "stands" atop the piezo stacks, each with a plate and screw on the top. Removing the screw and plate on each, the stands can be shifted together or apart to create an appropriate gap for the sample to bridge (the goal is to leave room to wrap a coil, but also support the sample). Once they're placed correctly, the screws can be replaced and some GE varnish can be dotted to keep them in place. Once they're glued in place, the screws can be removed.

To prepare the screws for when the sample is mounted, one should choose a

[3] https://razorbillinstruments.com/fc1x0-high-performance-cryogenic-stress-cell/

suitable number of washers such that the sample is not being compressed by the screws, but the plates the screws are holding down are close enough to have a thin layer of glue between them and the sample. For example, a 0.5mm thick sample should motivate the use of about 0.55-0.6mm of washers, so there's some small volume between the plate and the sample.

Now that the stands are placed correctly, the sample can be placed to bridge the gap. The heavy-duty glue of your choice (e.g., Stycast) should be added at this step, just to keep the sample attached to each stand. *Do not put too much! It will interfere with coil wrapping, which is already hard enough.* The glue should fill the volume between the bottom of the stand and the top plate, less the sample of course. Air pockets are not recommended. It is advisable to keep the screw (but not the top plate) in the hole with the washers, so the volume can be filled in. After one side is glued, the top plate can be placed and screwed in (not too tightly - just until snug. The purpose of the screws is not to keep everything held together long term; that's a job for the glue), and the process can be repeated for the other side. When finished, you should have something that ressembles Fig. **??**.

After the sample is glued down, the last step (if you're using Stycast, anyways. Check for your specific glue) is to cure it. For Stycast, we baked it at 60°C for a couple of hours. If you're following the same process, it is a good idea to cure the leftover Stycast with the sample, to have an idea of what the end product is, if it cured properly, *et cetera*. Stycast, for example, should end up glassy and hard.

Now, the sample mounting should be complete. That crystal isn't going anywhere!

2.1.3 Wrapping the Coil

If you're planning on doing NMR with your sample, you'll need a coil around it. This is as simple as it sounds, but deserves a section. Choose some thin insulated wire *which can handle the power you're going to put through it*. Cut it longer than you need for a few loops (pictured are 6 or 7) and leads. Use either your fingers or some tweezers to handle only the ends of the wire, and make a nice solenoid around your sample, leaving ample wire for both leads (more than you think you need). It might be helpful to use plastic or rubber-tipped tweezers to hold the coil in place as you wrap it.

When finished, visually inspect, under microscope if necessary, the coil to ensure there are no tears in the insulation; if you only handled the ends and checked the wire before starting, it should be pristine. If not, start again. Once the coil is wrapped without damage, use a blade of some kind to pull the insulation off of the leads, cutting excess wire off and leaving enough to have rounded solder points onto the stick's connection points, and enough material for thermal contraction (i.e., no taut wires or sharp bends). After all your work is complete, take some pictures for your family. It should look similar to Fig. **??**.

2.1.4 Soldering

The cables for RF signal you threaded through the strain cell earlier should be soldered onto each end of the coil. Ensure that there is ample "extra" wire such that cooling down to cryogenic temperatures does not introduce strain, or pull the wires into awkward positions. Further, the connections should be as continuously-shaped as possible to avoid high voltage/high frequency issues. The RF input polarity shouldn't matter; if it does, the reader is responsible for knowing this about their own experiment and acting accordingly. As such, choose whichever lead to solder to for maximum convenience. Keep in mind the leads are likely only exposed on the tips, as there are multiple layers of insulation. Once soldered, it is recommended to check continuity and resistance, both near the sample and from the coaxial interface at the head of the stick.

2.1.5 Bump Guard

The bump guard is rather straightforward to install. First, if detached, screw on the PPMS puck interface to the guard. The strain cell has slots on two edges. The screws (if in) should be removed from these, and the guard can be slotted in. Replace the four screws, and you're done.

2.1.6 Thermometer and Aux. Devices (Puck Interface)

Auxiliary devices, such as a thermometer, should be screwed onto the head of the strain cell in a manner that makes sense for the device and your sample. For example, a thermometer should be placed near to the sample. There are three connectors available on the puck interface (on the bottom, or samplefacing, side of the interface. See Fig. **??**) which devices can be plugged into. It does not matter which device plugs in to which connector.

2.1.7 Insertion to the PPMS

The PPMS should be brought to zero field and atmospheric temperature and pressure before beginning any of this. After removing any cap or device from the PPMS, the stick can be dropped (*figuratively*) in. Since the stick is very slightly too long, it may be necessary to pull the collar just below the head down to meet the PPMS port. Add the clamp back onto the connection between the PPMS and the stick's collar.

Now that the stick's in, you can begin making electrical connections. The stick has three ports, as well as the puck interface. One can wire the ports as they wish. The puck interface has a single tab on it, which is meant to slot into the bottom of the PPMS with a predetermined orientation. At our facility, this tab is roughly aligned with the front side of the PPMS.

2.2 Strain Cell Capacitance Measurement Setup

The strain cell's capacitance can be measured using an AD7745 capacitance to digital converter (CDC) and an Arduino Nano (or similar). Code exists TODO: where. In the referenced repository, documentation exists to describe all the soft-, firm-, and hardware interfaces. This piece of hardware utilises the I2C Protocol[4], and it is advised that pull-up resistors are added to the system if none are pre-installed on the microcontroller used. Arduino Nanos do *not* come with pre-installed pull-up resistors! In experiment, we used as low as 900Ω resistors for both the clock (SCL) and data (SDA) lines.

2.3 SCOUT Setup

The Tecmag SCOUT system is fairly straightforward to set up, since it's mostly pre-assembled. All you need to do as the user is choose an appropriate transcoupler and preamplifier for your frequencies. This is quite literally as simple as picking the ones that list your frequency, and hooking them up as in Fig. **??**. Then, you can plug the probe cable into the stick. It is sometimes nice to have a multiplexer such that a network analyser or the SCOUT can

[4] https://www.ti.com/lit/pdf/sbaa565

be connected to the stick - if you're going to do this, **ensure that the SCOUT output is *not* connected to the network analyser in any way, whatsoever!**

3 Hardware Testing

3.1 The Stick

The stick, for the most part, is only really testable for continuity in each of the connections. Individual auxiliary devices and the strain cell can be tested for dummy data acquisition depending on the device. Once in the PPMS (or, I suppose, even before), the tuning can be inspected and/or adjusted via a network analyser connection.

3.2 SCOUT

The SCOUT system can be tested in isolation with some zero-field samples, as in Fig. **??**. Once a signal is found from one of these, the system is likely working. Apart from this, please contact Tecmag support or consult your user manual.

4 Software Setup

4.1 The Stick

Really, this section runs through the configuration of the puck, as the rest doesn't require software. Assuming this is to take place in a NICOS instance, the computer connected to the PPMS should be running a Frappy [**?**] server. Find its IP address and enter it into the `frappy_ppms` and/or `frappy_stick` (possibly `frappy_main`) module in NICOS. The PPMS should connect and you should see many submodules appear, such as `se_tt` (PPMS temperature setpoint), `se_mf` (PPMS magnetic field), *et cetera*. The puck connections will appear as `se_rX` where `X` denotes an integer. The value itself represents a resistance (in Ω), and the PPMS must be configured to apply a constant voltage over/current through the connections to read values with meaning. This is done in PPMS MultiVu, and possibly using the switchboard to route connections from the power supply (managed in software via PPMS MultiVu) to the actual PPMS hardware (puck).

4.1.1 Thermometer Calibration

The thermometry on the PPMS itself is quite reliable, though due to the high thermal mass of the strain cell, we include a small on-stick thermometer to give more accurate results for the sample temperature. This sensor is a Cernox thermocouple, specifically a Cernox 1030-SD[5] (high-temperature and high-vacuum packaging). Calibration should be performed to convert resistance values to temperatures.

4.2 SCOUT

Please refer to the software documentation[6] (origin - NMR Spectroscopy PSI[7]) for a comprehensive guide to setting up the software interface for the SCOUT.

[5] https://www.lakeshore.com/docs/default-source/product-downloads/catalog/lstc_cernox_l.pdf?sfvrsn=41b96c23_4
[6] https://www.psi.ch/media/103166/download
[7] https://www.psi.ch/en/lin/nmr-spectroscopy