

EPR Operating Guidelines – Sample Preparation

Solid Powder Sample:

If you have your sample in a vial, easiest way to get it into the EPR tube would be to use the tube like a scoop and tap the sample to the bottom. 1-2 cm of sample inside the tube is more than sufficient.

If the sample is not in a vial, you can fold a piece of weighing paper diagonally and use it as a slide to get the powder into the tube.

To clean a tube after solid powder sample, use a fuzzy tube cleaner and a bit of quick drying liquid, such as Methanol. Use a can of compressed gas to blow out what's left.

Solution Sample:

Low Dielectric Solvent (below 8):

Use a pipette to transfer your sample to a normal EPR tube. 50uL – 100uL of sample inside the tube is sufficient.

Clean the tube by pouring out the solution to an appropriate container and washing the tube with an applicable solvent. Blow dry with a can of compressed gas.

High Dielectric Solvent (above 8): See aqueous sample guidelines

Aqueous Sample:

Use the flat cell resonator. The instructions below are copied from the spectra acquisition procedures:

The Flat Cell Resonator is designed to allow a minimal surface area of your sample to be exposed to the magnetic field so that the signal acquired may be of higher resolution. It makes obtaining spectra from samples with high dielectric solvents possible. Aqueous solutions are included in that list.

1. The Flat Cell is only compatible with the 4102ST resonator. The resonator should be gold plated. Ask the facility manager to help you switch resonators if necessary.
2. Obtain the Flat Cell Resonator from the facility manager.
3. Ensure that the glass stopper is inserted into the end which aligns it through the collet direction, as in the tip of the glass stopper points to the unperforated, thinner part of the white collet.
4. Put the machine in **Stand By** mode and physically remove the collet currently in the resonator by unscrewing the black collar and pulling the white collet out. Do not replace the collar yet.
5. Fill the flat cell resonator using a pipette. Draw your sample throughout the entire area of the rectangle by gently pulling out the glass stopper, very slightly to release pressure.
6. Wipe clean the outside of the flat cell resonator. Insert it into the resonator gently.
7. Align the resonator so that the edge, rather than the face, of the flat cell is aligned with the magnetic field. This means if you are to stand in front of the resonator, you will face the large surface, not the edge.
8. Replace the collar and begin tightening. Before completely tightening, pull the flat cell resonator up so that the entirety of the smoked glass part is above the collar and collet. Tighten fully.
9. Put the machine into **Tune** mode and follow tuning procedures.

10. When the spectrum has been acquired to satisfaction, put the machine into **Tune** mode, remove the collar, and remove the flat cell with the collet. Drain the sample by removing the glass stopper and draw out any remaining sample using a paper towel. Use water and methanol to clean the interiors and blow dry with a can of compressed air. Let air dry if necessary.
11. Return the Flat Cell to the facility manager when done.