NMR spectroscopy

Sample preparation:

Before discussing sample preparation, there are a few points about the nmr tube that need to be mentioned.

1. The tube is quite fragile. It is precision made from thin glass to specific dimensional tolerances. Because of the thin glass, it may break very easily. Abusive handling of the tube is therefore to be avoided. Take special care in removing and replacing the tube cap.
2. The bottom of the nmr tube should be handled as little as possible. This is not because the tube is fragile, but that your hands are quite dirty (from an nmr point of view). Excessively dirty hands make for dirty tubes, which makes for a dirty instrument, and poorer resolution in your spectra.
3. Before preparing a new sample, wet a Kimwipe with acetone and clean the bottom of your nmr tube. Afterwards, handle the tube by the top only.

There are differences in sample preparation depending upon whether the sample is a liquid or a solid.

Liquid samples:

Add close to 1mL of deuterated solvent to a sample vial. Remember that chlorinated solvents are suspected carcinogens, so use a fume hood. Add a few drops of the liquid sample and thoroughly mix. Transfer this to the nmr tube through a disposable pipette that has been lightly plugged with a small wad of Kimwipe. The routine filtering of samples is a good way to ensure that the resolution of the nmr experiment is maintained.

Solid samples:

The most important issue here is sample mass. An organic sample has an appreciable part of its mass in H or C. An inorganic sample is mostly metal, so it is important to add more sample material!

For the typical organic sample: Measure out approximately 40 - 60 mg of the compound to be run and place it in the vial. The weights listed are guides only. If your sample has few protons per molecule, or you are recording a ^{13}C spectrum, you will need to use more material. Use a balance if necessary, since it is difficult to estimate the weights of some solids by eye.

For the typical inorganic sample: Add to the vial approximately 1 mL of an appropriate deuterated solvent. Add as much solid as possible to saturate the solution. (Remember that nmr samples can be recovered for subsequent steps, or for recording ir spectra.) Swirl, or stir, to dissolve the compound. If there is a lot of suspended material, allow it to settle and transfer the supernatant by pipette. Put the supernatant in to the nmr tube through a disposable pipette that has been lightly plugged with a small wad of Kimwipe. The routine filtering of samples is a good way to ensure that the resolution of the nmr experiment is maintained. If necessary rinse the filter with a small amount of nmr solvent. Avoid filling the nmr tube with more than 4 cm of liquid. The additional weight may cause problems with the spinning and hence, the resolution will degrade.