

LC-MS Sample Preparation Protocol

Use only HPLC grade solvents

- Dissolve a small amount of sample in H₂O or CH₃CN/H₂O mixture (Buffer A and Buffer B)
- If particulates are visible, centrifuge mixture and filter through 0.22µm insert
- A minimum volume of 50µL should be added to the HPLC sample vial for analysis

For peptides (crude or HPLC purified), the following procedure must be followed prior to analysis

- After peptide cleavage and ether extraction, vacuum dry or lyophilize the sample
- Prepare the sample according to the procedure above (make sure to filter out any salts or particulates)
- Maintain a concentration in the micromolar range

For small molecules, no crude sample may be analyzed directly from a reaction mixture. The sample must undergo a work-up prior to analysis.

- Remove a small aliquot from reaction mixture, dissolve in water and extract with solvent (depends on conditions)
- Vacuum dry organic phase and redissolve in a mixture of Buffer A and Buffer B
- Filter the sample and add a minimum volume of 50µL to HPLC sample vial for analysis