**LCMS Sample Preparation**

For Crude/Pure Products (solvent free sample after work-up):

1. Dilute sample and dissolve in **50 %** **LCMS-Grade acetonitrile and 50% LCMS-Grade water** to a concentration of **~0.2 mg/mL**.
2. **ALWAYS** **filter** through yellow syringe filter, even if the sample looks fully dissolved.
3. If the sample does not dissolve, then DO NOT SUBMIT to the LCMS or try and re-filter. Submit your sample for NMR instead.
4. At the end of your sequence table, always **add a wash injection**. There should be a vial containing methanol in P1-F11. Check it is full enough and make sure to run a 5-100% acetonitrile method, such as the EGT or WASH methods.

Reaction Monitoring (reaction mixture before work-up):

1. Pipette a drop of your reaction mixture and place in a small vial.
2. Dilute with an appropriate organic solvent from the original reaction procedure.
3. Perform a mini-workup: Quench the reaction mixture according to the procedure e.g. quench strong acids with a base, quench strong bases with an acid, filter metals (e.g. Pd) through Celite, perform any wash/extraction.
4. Remove solvent *in-vacuo*.
5. Treat sample as above.

Checking Fractions following Purification via Column Chromatography:

1. Identify relevant fractions using TLC and combine.
2. Remove solvent from relevant fractions (Except for fractions from reverse phase columns done in Methanol/Water – 1 drop can be directly placed into LCMS vial, diluted with LCMS-Grade acetonitrile and water then filtered).
3. Treat sample as above.

Extra Points:

* Always run perform TLC analysis of your reaction before submitting an LCMS.
* For any questions, refer to **Eve, Yinuo or Yuhang**.