# EXPERIMENT 4: Synthesis of Schiff base ligands

In this experiment, you will synthesize a ligand using a simple condensation reaction between an aldehyde and an amine. The product of this reaction is often referred to as a Schiff base. Schiff base ligands provide a simple and effective way to investigate the reactivity of a complex by altering substituents on the aldehyde or amine reactant.

**Safety considerations:**

Ethylenediamine is a strongly alkaline, caustic material. It has a strong ammonia-like odor that is very irritating. It must be used in the hood at all times. Exercise special care when using ethylenediamine and be sure to promptly clean up any spills or splashes using plenty of soap and water.

## Synthetic Procedures:

### A. Synthesis of SALEN-H2



1. Weigh 0.8 g of salicylaldehyde into a 50 mL Erlenmeyer flask and add ~15 mL of 95% ethanol and a magnetic stirring bar.  Bring this solution to a gentle boil while stirring using a hot plate-stirrer (setting 1-2).

2. Using a small syringe, add 0.5 equivalents of ethylenediamine dropwise to the heated solution.  After addition is complete, continue to stir and reflux the solution for approximately 10 minutes, then remove the flask from the hot plate, and allow it to cool to room temperature.

3. Cool the flask in an ice/water bath.  Collect the resulting bright yellow flaky crystals by vacuum filtration.

4. Wash the crystals with 2-4 mL of cold ethanol, then air dry the product.

5. Determine the yield (in grams and %) and melting point of the product.

6. Characterize the product by 1H-NMR and FTIR spectroscopies.

7. This ligand will be used in the functionalization of a Vanadium Complex.

### B. Synthesis of SOAP Ligands



1. Weigh 0.80 g of 2-aminophenol into a 25 mL round-bottomed flask and add 8 mL of 95% Ethanol, 5 mL of DI water, and a magnetic stirring bar. Bring the solution to a gentle boil while stirring using a hot plate-stirrer.

2. Add one equivalent of salicylaldehyde, and 3-4 drops of acetic acid to the heated solution.

3. Continue to heat gently for ~25 minutes, then remove the flask from the hot plate, and allow it to cool to room temperature.

4. Cool the flask in an ice/water bath. Collect the resulting crystals by vacuum filtration.

5. Wash the crystals with 2-4 mL of cold water, then air dry the product.

6. Determine the yield (in grams and %) and melting point of the product.

7. Characterize the product by 1H-NMR and FTIR spectroscopies.

8. This ligand will be used in the synthesis of a fluorescent aluminum complex.

NOTE: There is no full report for this experiment, as these ligands will be used for upcoming experiments.