#### Aluminum Chloride (small scale)

#### Materials needed:

- 1. Anhydrous Aluminum Chloride
- 2. Calcium Chloride
- 3. Sodium Hydroxide
- 4. Magnesium Sulfate
- 5. DCM

### **Reaction Set Up:**



#### **OPERATING PROCEDURE:**

- 1. Dry your CBD isolate to be used for 24 hours in a vacuum oven at 125F
- 2. Mix your CBD with a 2:1 ratio DCM:CBD
- 3. Allow this solution to sit in calcium chloride (10%) for 4 hours. Remove calcium chloride.
- 4. Run a stream of nitrogen through this solution for 6 hours.
- 5. Add 5% alcl3 to be used during the reaction (by weight of CBD) to your addition funnel in a glove box. Transfer over to your reaction apparatus.
- 6. Add CBD solution to apparatus.

- 7. Vacuum and backfill your apparatus 3x with nitrogen. Allow nitrogen to bubble through the reaction for the duration of the reaction.
- 8. Set stir bar to 400 rpms
- 9. Drop the temperature of reaction incrementally to -5C
- 10. Add your alcl3 slowly. If exothermic spike reaches passed 0C stop addition and await temperature stabilization.
- 11. Once all alcl3 is added wait for 30 minutes.
- 12. Reaction should be complete.

### Quench:

- 1. Add 15% sodium hydroxide in water at a volume of 1:1 reaction solution: aq lye
- 2. Keep bubbling nitrogen throughout the duration of the quench
- 3. Add .5 volumetric liters of distilled water
- 4. Remove chilling and warm the reaction to room temperature, stirring rapidly the entire time.
- 5. Add 10% weight of the alcl3 used during reaction (magnesium sulfate)

### Filtration:

1. Filter through 2.5 micron all of the precipitates created during the quench.

## WASHES:

# ALL LLE WATER IS USED AT A 1:1 VOLUMETRIC RATIO OF OIL/SOLVENT:WATER

- 1. Saturated brine wash. Mix vigorously, allow to separate and discharge aqueous
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- 3. 1% Citric acid water. Mix vigorously, allow to separate and discharge aqueous
- 4. 5% sodium bicarbonate wash. Mix vigorously, allow to separate and discharge aqueous
- 5. As many distilled water washes until the PH is reading 6.5-7