

20.7 g of o-nitroaniline
makes ~ 28 g
of Grieco reagent



Add 20.7 g o-nitroaniline to large round bottom equipped with a large stir bar (or a three-neck with mechanical stirring)



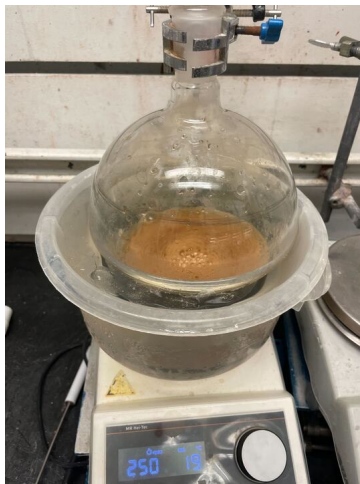
Add 25 mL 6 M HCl and stir for 5 minutes before cooling to 0°C



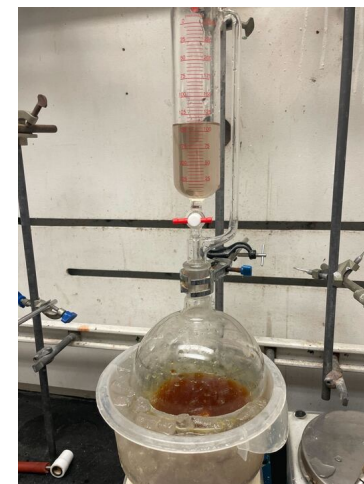
With an addition funnel, slowly add a solution of 12.4 sodium nitrite in 60 mL water



After stirring for 20 minutes, add 3.5 g urea to the reaction mixture



Add 25 g sodium acetate (should be ~ pH 6)



Slowly add a solution of 21.6 g potassium selenocyanate in 100 mL water (results in a "mushy dark brown solid")



Collect the solid and rinse with water



Recrystallize from 700 mL ethanol



Filter and collect purified Grieco reagent!