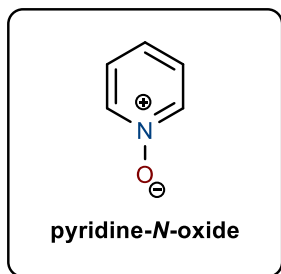
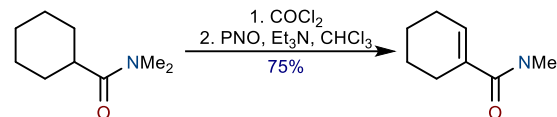
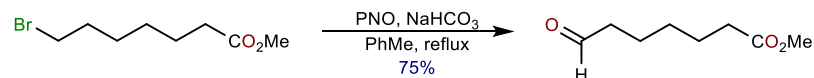
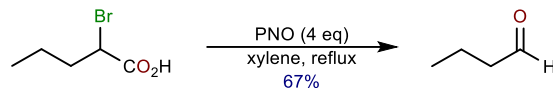
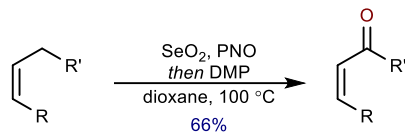


Background



A useful reagent for oxidations...



- PNO is extremely hygroscopic
- Purified dry material is a white solid
- Depending on moisture content, wet material can be anything from a brown solid to a yellow liquid

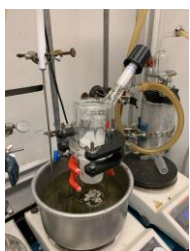
Qin, T. *Nat. Synth.* **2023**. <https://10.1038/s44160-023-00437-w>

Kilényi, S. and Mousseau, *e-EROS*. **2015**, <https://doi.org/10.1002/047084289X.rp283.pub2>

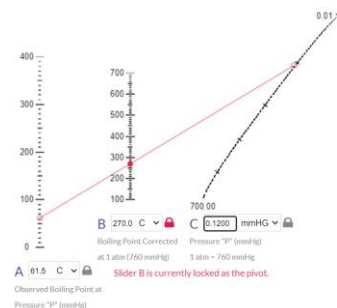
Purification from a solid (vacuum sublimation)



1. Gather pieces for vacuum sublimation
2. Weigh out PNO accounting for some water content
3. Place wet PNO into vacuum sublimator outer casing
4. Assemble vacuum sublimator and clamp. Ensure the tap is closed, attach the vacuum line and open the vacuum. Slowly open the tap to place the entire system under vacuum.



5. Place your cooling bath into the cold finger (dry ice/acetone pictured) and lower the sublimator into the heating bath.



6. Use the Sigma nomograph to determine your heating temperature. E.g. PNO bp (760 torr) 270 °C, vacuum measured at 0.12 torr, so bp lowered to 61.5 °C. Stir plate set to 65 °C.

<https://www.sigmaaldrich.com/US/en/support/calculators-and-apps/pressure-temperature-nomograph-interactive-tool>

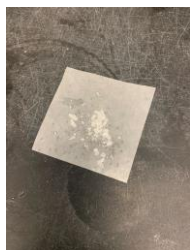
Purification from a solid continued



7. When all the material has sublimed you should see a white solid on the bottom of the cold finger. Remove the apparatus from heat and let the cold finger warm to room temp BEFORE removing the vacuum (otherwise condensation will collect and you will be left with wet material again!)



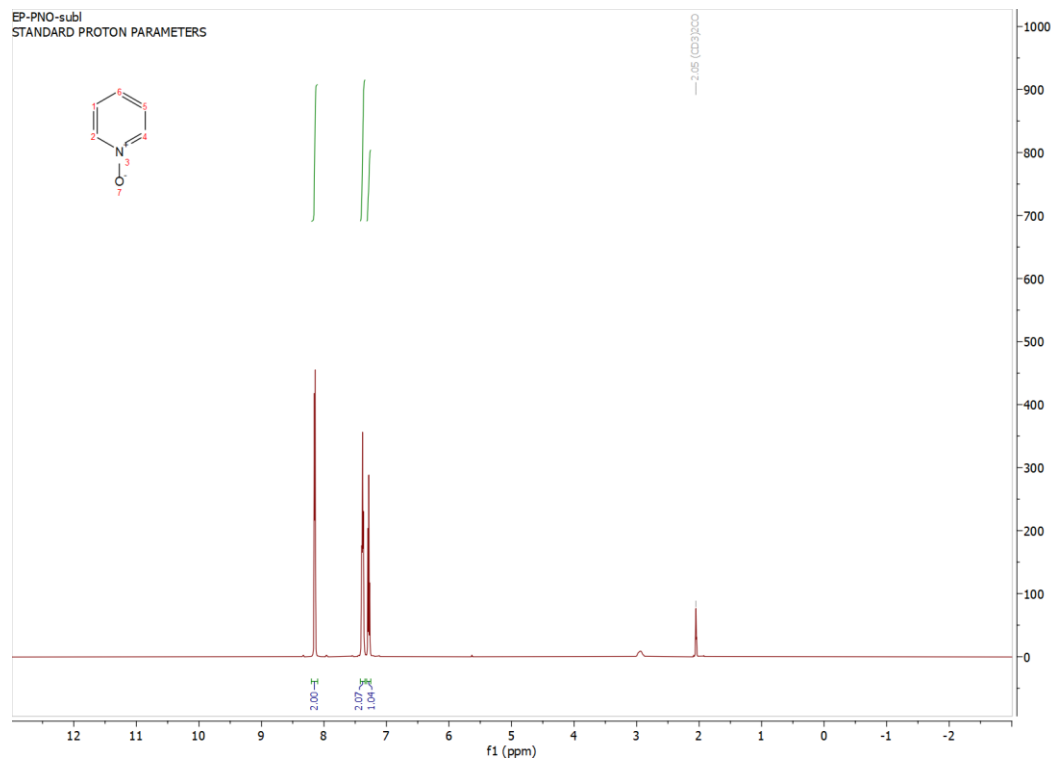
8. After warming to room temp, flush the apparatus with nitrogen and carefully remove the cold finger with the sublimed PNO



9. Purified PNO!



10. Flame/oven dry two flasks and a U-shaped vacuum connector. Place PNO in one flask and P₂O₅ in the other and dry under high vacuum for at least 12 hours



Purification from a liquid

I have found that dissolving liquid PNO in DCM followed by removal of the solvent by rotary evaporation and drying on a high vac line for 30 mins results in the formation of an amorphous yellow solid that can be vacuum sublimed according to the above protocol to give pure material.