Standard Operating Procedure

Read the EH&S Standard Operating Procedures Fact Sheet before filling out this form. Print out the completed form and keep a readily accessible hard copy in the lab (also keeping an electronic copy is highly recommended).

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SOP Title: Handling of Nitric Oxide Gas

Principal Investigator: Robert G. Bergman

Room and Building: 675 Tan Hall

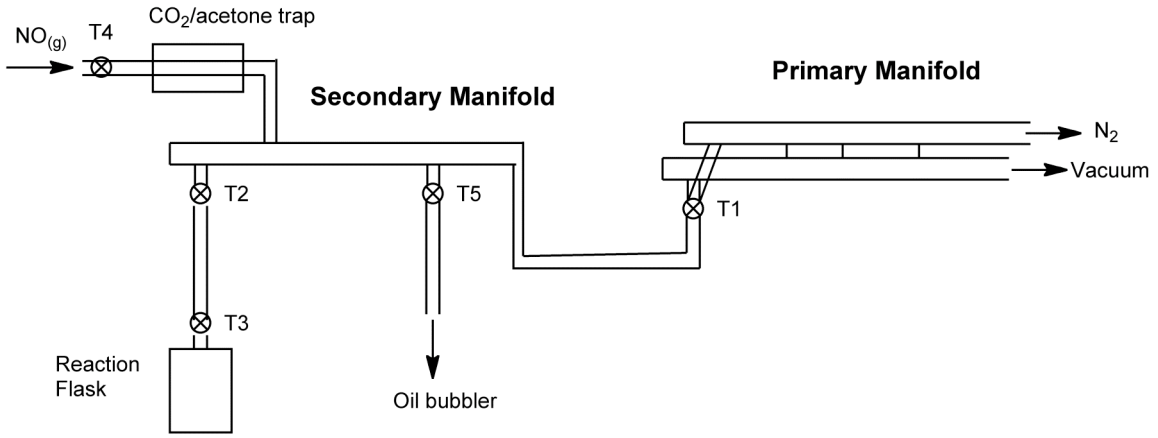
Lab Phone Number: 510-642-1548

Section 1 – Process

The following represents a generalized procedure for use of nitric oxide gas in a closed system for introduction to Schlenk tubes, J. Young NMR tubes, or round-bottomed flasks.

Nitric oxide must be stored in a gas cabinet, and reactions utilizing it must be run in a well-ventilated fume hood

A secondary manifold is required for the safe handling of NO (g). In addition, NO may be purified by passage through a -78 ^{o}C trap. The following setup has proven useful for the manipulation of this gas with minimum exposure to the user.



Weigh reagents and solvent into the required reaction vessel (round-bottomed flask, NMR tube or Schlenk tube), setting up the reaction mixture under an inert atmosphere if required. Attach the desired reaction vessel to the secondary manifold by evacuating and backfilling up to the tap of the flask using T1 and T2, keeping T3, T4 and T5 closed.

At this point the reaction mixture should be degassed *via* the freeze-pump-thaw technique (it should be noted that glassware under reduced pressure may implode and adequate blast protection should be used). Under a purge of inert gas (nitrogen or argon), open T3 to vacuum after freezing the reaction mixture in liquid nitrogen, seal the flask at T3 (under static vacuum) and warm to room temperature. Once at ambient temperature, freeze the mixture again, open T3 to vacuum and evacuate the flask. Close T3 and warm to room temperature. Repeat this last procedure, leaving T3 closed once the flask is warmed to room temperature.

Introduce NO into the manifold by closing T1 and opening T4 (note that at this point T5 and T3 should be closed and T2 open).

Extreme care should be taken to keep the pressure of NO at approximately 1 atm.

The user should be aware that the apparatus is being pressurized to the cylinder pressure and be aware of the pressure setting on the NO tank. The manifold should be left open to NO(g) by leaving T1 closed and T4 open.

Introduce NO to the reaction mixture by opening T2 and allowing the gas to diffuse in to the reaction solvent. At this point the pressure may be increased to the desired level or, for long reaction times requiring an open system, T3 may be opened, allowing NO to bubble through the system. After the required time, close T3 and T4 and remove the remaining NO from the manifold under reduced pressure by opening T1.

Monitor the reaction as desired and, once it is complete, remove the reaction vessel from the secondary manifold. Reactions run in NMR tubes may be sealed and removed from the line as desired.

The -78 ^{o}C trap should be dismantled and cleaned before each new reaction. Furthermore, care should be taken when taking down the primary manifold following the reaction as, during the purging of the secondary manifold NO(g) may condenses in the liquid N_2 trap.

Section 2 – Hazardous Chemicals

Nitric oxide is highly toxic with a lethal concentration of about 100 ppm. Other hazardous chemicals may be present in the reaction mixture, depending on the experiment being performed.

Section 3 – Potential Hazards

The primary hazard is nitric oxide, which is highly toxic by inhalation as mentioned above. Other reagents in the reaction mixture may also be toxic or flammable, as may the solvent. Glassware may explode or implode under pressure or vacuum, respectively; care should be taken that all flasks and tubes are free of cracks and that they are not allowed to become overpressurized.

Section 4 – Approvals Required

Nitric oxide use in fume hood H3 in 674 Tan Hall is approved by EH&S on the conditions of the approval documentation agreed upon by Phil Maynard and Mark Crimmin. New users should not perform this experiment without supervision. For experienced users, it is not necessary to obtain approval each time an experiment of this sort is performed routinely, but as it is still a hazardous

procedure, it is not an appropriate experiment to perform when you are the only person in the lab.

Section 5 – Designated Area

The reaction must be performed in a well-ventilated fume hood with a face velocity of approximately 100 feet/min. NO(g) must be stored and secured in a gas cabinet with a minimum face velocity of 200 feet/min.

Section 6 – Special Handling Procedures and Storage Requirements

See Section 5 above.

Section 7 – Personal Protective Equipment

Thick goggles, fire-resistant gloves and a fire-resistant lab coat should be worn at all times during this procedure, and the hood sash should be closed and fully pulled down whenever possible.

Section 8 – Engineering/Ventilation Controls

It is essential that the fume hood used be operating well with a sash that moves smoothly and does not stick. If the experiment is set up in a glove box, it should be conscientiously maintained at appropriately low water and oxygen levels.

Section 9 – Spill and Accident Procedures

A minor NO leak should be countered by a considered response of isolating the leak and ensuring ventilation of the affected areas. A major NO leak requires immediate evacuation of the affected area. NO leaks can often be detected by the observation of brown NO_2 gas, which NO forms when it reacts with atmospheric oxygen.

Section 10 – Waste Disposal

Appropriate disposal of the reagents and solvents used will depend on the substances present in the reaction mixture. If any pyrophoric materials are present, they should be carefully quenched as per the SOP for quenching pyrophorics.

Section 11 - Decontamination

Nitric oxide leaks should be isolated, and the area allowed to ventilate. Spills of organic solvents should be washed with acetone and the waste disposed of in a suitably labeled container.

Section 12 – Process Steps

Process Steps	Safety Measures
1. Weigh reagents into reaction vessel (round-bottomed flask, Schlenk tube or J. Young NMR tube).	

2. Attach reaction flask to secondary manifold	
3. Freeze-pump-thaw reaction mixture	Make sure hood is fully closed, and ensure that no leaks
4. Isolate reaction mixture	
5. Purge secondary manifold	
6. Introduce NO to reaction flask	Ensure that the fume hood is well ventilated, and be watchful for any leak of NO gas (visible as brown NO_2).
7. Remove NO from secondary manifold under reduced pressure	

Training Documentation

Name (Printed)	Signature	Date