**STANDARD OPERATION PROCEDURE FOR CVD NANOTUBE REACTOR (Reactor #1 in Lab D-102)**

Date of the last revision: 6/ 14/ 12

**Last reviewed by:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_Date:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_**

**SOP discussed with:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_Date:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_**

**Personal Protective Equipment (PPE) Required:** Safety glasses, lab coat and gloves.

(Preparation of the reaction feedstock has to be performed before starting the reaction inside the fume hood).

**Chemicals used (IUPAC Name, CAS Number, MSDS, Associated Risks**

**Nitrogen** – Nitrogen, CAS Number:8013-59-0, Contact with rapidly expanding gas may cause burns or frostbite. Can act as a asphyxiant. **Hydrogen** –Hydrogen, CAS Number: 4368-28-9, Flammable. Contact with rapidly expanding gas may cause burns or frostbite. Can act as a asphyxiant. Acute or chronic respiratory conditions may be aggravated by overexposure to this gas.

**Ethylene** –Ethene, CAS Number: 74-85-1, MSDS #: 001022 Flammable. Contact with rapidly expanding gas may cause burns or frostbite. Can act as a asphyxiant. Acute or chronic respiratory conditions may be aggravated by overexposure to this gas

**Isopropanol** –Isopropyl alcohol,CAS Number: 67-63-0, Highly flammable. If inhaled can cause respiratory tract irritation. Vapors may cause drowsiness and dizziness. Causes skin and eye irritation.

**Iron nitrate** –Iron (III) nitrate,CAS Number: 7782-61-8, May intensify fire, oxidizer, harmful is swallowed. Causes skin irritation and serious eye irritation. May cause respiratory irritation.

**Cobalt nitrate** –Cobalt (II) nitrate hexahydrate, CAS Number: 10026-22-9, Strong oxidizer, emits nitrous oxides when heated to decomposition, incompatible with ozidizable substances (paper, wood, sulfur, aluminum, plastics).

**Aluminum nitrate nonahydrate** – Aluminum nitrate nonahydrate, CAS Numberl: 7784-27-2, May be harmful if swallowed, causes skin irritation, and causes serious eye irritation. Materials to avoid with are: strong reducing agents, powdered metals, and strong acids. Hazardous decomposition products formed under fire conditions.

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| **1. - REACTOR VALVE CHECKLIST** | | |  |  |
| 1.1 | | Turn valve COV1 so that it will point left. |  |  |
| 1.2 | | Turn valve EV1 so that it will point down. |  |  |
| 1.3 | | Turn valve NV1 so that it will point down. |  |  |
| 1.4 | | Turn valve HV1 so that it will point down. |  |  |
| 1.5 | | Turn valve NV2 so that it will point to the left. |  |  |
| 1.6 | | Turn valve NV3 so that it will point down. |  |  |
| 1.7 | | Turn valve COV2 so that it will point down. |  |  |
| 1.8 | | Turn valve COV3 so that it will point down. |  |  |
| 1.9 | | Turn valve HV2 so that it points to the left. |  |  |
| 1.10 | | Turn valve NV4 so that it points to the left. |  |  |
| 1.11 | | Turn valve EV2 so that it will point down. |  |  |
| 1.12 | | Turn valve EV3 so that it will point down. |  |  |
| 1.13 | | Turn valve NV5 so that it will point to the left. |  |  |
| 1.14 | | Turn valve EV4 so that it will point up. |  |  |
| 1.15 | | Turn valve EV5 so that it will point up. |  |  |
| 1.16 | | Turn valve RVV so that it will point to the left. |  |  |
| 1.17 | | Turn valve RVV1 so that it will point up. |  |  |
| **2. - PLACING SUBSTRATE IN THE REACTOR VESSEL** | | |  |  |
| 2.1 | | Inspect quartz glass tube for cracks. |  |  |
| 2.2 | | Place the substrate in the glass reactor vessel. |  |  |
| 2.3 | | Place the glass reactor vessel in the reactor heating unit. | Glass reactor vessel is made of glass and can break easily. | Handle with care. |
| **3. – OPENING THE TANKS** | | |  |  |
| 3.1 | Go to the gas tanks on the wall, opposite of the wall of the reactors. | |  |  |
| 3.2 | Open valve NTV2, point to the right. | |  |  |
| 3.3 | Open valve NTV4 so that it points up. | |  |  |
| 3.4 | Keep valve NTV3 pointing towards the wall. | |  |  |
| 3.5 | Open the nitrogen tank valve NTV1. | |  |  |
| 3.6 | Set the pressure on gauge NTP2 to 20 psi using valve NTV5. | |  |  |
| 3.7 | Open valve NTV6 to allow gas to flow to the reactor. | |  |  |
| 3.8 | Open valve HTV2, point to the left. | |  |  |
| 3.9 | Open valve HTV4 so that it points up. | |  |  |
| 3.10 | Keep valve HTV3 pointing towards the wall. | |  |  |
| 3.11 | Open the hydrogen tank valve HTV1. | |  |  |
| 3.12 | Set the pressure on gauge HTP2 to 20 psi using valve HTV5. | |  |  |
| 3.13 | Open valve HTV6 to allow gas to flow to the reactor. | |  |  |
| 3.14 | Open valve ETV2, point to the right. | |  |  |
| 3.15 | Keep valve ETV3 pointing towards the wall. | |  |  |
| 3.16 | Open valve ETV4 so that it points up. | |  |  |
| 3.17 | Open the ethylene tank valve ETV1. | |  |  |
| 3.18 | Set the pressure on gauge ETP2 to 20 psi using valve ETV5. | |  |  |
| 3.19 | Open valve ETV6 to allow gas to flow to the reactor. | |  |  |
| **4. - PURGE WITH NITROGEN AND CHECKING FOR A LEAK** | | |  |  |
| 4.1 | | Turn on power strip, temperature controllers, and mass flow controller. |  |  |
| 4.2 | | Turn valve NV to the left to allow nitrogen to flow to the fume hood. |  |  |
| 4.3 | | Set the nitrogen mass flow (Channel 1) to 30% (300 cm3/min), using the Porter mass flow controller unit. |  |  |
| 4.4 | | Once the nitrogen flow has stabilized, point valve NV to the right, to allow the nitrogen gas to flow into the reactor vessel. |  |  |
| 4.5 | | Allow nitrogen to flow through the reactor for about 3 minutes. |  |  |
| 4.6 | | Close valve, RVV1. Point towards the left. This will trap the gas in the reactor vessel. |  |  |
| 4.7 | | Watch the pressure gauge above the reactor vessel and wait for it to reach 10 psi. Immediately turn valve RVV to the right. Set the nitrogen mass flow to 0.0%, using the Porter mass flow controller unit. | Glass vessel is under pressure. | Watch pressure carefully. |
| 4.8 | | Then wait for about 5 minutes to see if the pressure drops. If the pressure drops then a leak is present. |  |  |
| 4.9 | | If no leak is present then proceed to the next step. |  |  |
| 4.10 | | Turn valve RVV1 so that it points up to allow the nitrogen gas to leave the reactor. | Opening the valve to quickly will cause the sample to move. | Open the valve slowly. |
| 4.11 | | Turn valve RVV to the left. |  |  |
| **5. - SETTING THE FURNACE** | | |  |  |
| 5.1 | | Hold the reset button until the top display shows “x” and the bottom “x”. |  |  |
| 5.2 | | Continue holding the reset button and use the arrow keys to scroll to “rSP9” on the top display. |  |  |
| 5.3 | | Set the thermostat to increase the temperature to 560°C, in 50 minutes. Then to hold at 560°C for 30 minutes. Set the thermostat to increase the temperature to 760°C, in 20 minutes. Then to hold at 760°C for 30 minutes. | Avoid burns, reactor oven set at 560°C - 760°C | Wear insulating gloves or use caution when touching parts of the reactor. |
| **6. - REDUCTION PROCEDURE** | | |  |  |
| 6.1 | | Turn valve NV to the left to allow nitrogen to flow to the fume hood. |  |  |
| 6.2 | | Set the nitrogen mass flow (Channel 1) to 30% (300 cm3/min), using the Porter mass flow controller unit. |  |  |
| 6.3 | | Point valve NV to the right, to allow the nitrogen gas to flow into the reactor vessel. Allow nitrogen to flow through the reactor for about 3 minutes. |  |  |
| 6.4 | | Open valve HV, point to the left, to allow the hydrogen gas to flow into the fume hood. |  |  |
| 6.5 | | Set the hydrogen mass flow (Channel 3) to 30% (150 cm3/min), using the Porter mass flow controller unit. |  |  |
| 6.6 | | Turn valve HV, point to the right, to allow the hydrogen gas to flow into the reactor vessel. Do this once the mass flow controller shows the flow of hydrogen has steadied. |  |  |
| 6.7 | | Press the “Up Arrow” on the temperature controller to start the furnace cycle. |  |  |
| 6.8 | | Point valve NV to the right and set nitrogen mass flow (Channel 1) to 0%, using the Porter mass flow controller unit. |  |  |
| 6.9 | | After 78 minutes. Proceed to the next step. |  |  |
| **7. – RAMP WITH NITROGEN** | | |  |  |
| 7.1 | | Turn valve NV to the left so that nitrogen can flow to the fume hood. |  |  |
| 7.2 | | Set the nitrogen mass flow (Channel 1) to 30% (300 cm3/min), using the Porter mass flow controller unit. |  |  |
| 7.3 | | After 80 minutes. Turn the valve HV to the left to divert the hydrogen to the fume hood. |  |  |
| 7.4 | | Turn valve NV to the right so that nitrogen can flow into the reactor. Do this once the mass flow controller shows the nitrogen flow has steadied. |  |  |
| 7.5 | | Set the hydrogen mass flow (Channel 3) to 0.0% using the Porter mass flow controller unit. |  |  |
| 7.6 | | Turn valve HV so that it points up. |  |  |
| 7.7 | | Allow nitrogen to run through system during the ramping procedure. |  |  |
| 7.8 | | After 18 minutes proceed to the next step. |  |  |
| 7.9 | | Once ramping is complete. Turn the NV valve to the left so that nitrogen gas will flow to the fume hood. |  |  |
| 7.10 | | Set the nitrogen mass flow to 0%, using the Porter mass flow controller unit. |  |  |
| 7.11 | | Turn valve NV so that is points up. |  |  |
| **8. – REACTION** | | |  |  |
| 8.1 | | Turn valve EV so that it points to the fume hood line. |  |  |
| 8.2 | | Set the ethylene mass flow (Channel 4) to 20% (200 cm3/min), using the Porter mass flow controller unit. |  |  |
| 8.3 | | After 20 minutes turn valve NV so that it points to the left. |  |  |
| 8.4 | | Turn valve EV so that it points to the right. This will allow the ethylene to flow into the reactor vessel. |  |  |
| 8.5 | | Set the nitrogen mass flow (Channel 1) to 0%, using the Porter mass flow controller unit. |  |  |
| 8.6 | | Turn valve NV so that is points up. |  |  |
| 8.7 | | After the reaction time is complete, 30 minutes. Proceed to the next part. |  |  |
| **9. – PURGING THE REACTOR AND VENTING THE GAS LINES CLOSING THE TANKS** | | |  |  |
| 9.1 | | Turn the valves HV and EV to the left so that the gas will vent to the fume hood. |  |  |
| 9.2 | | Turn the valve NV to the right so that it will go to the fume hood. |  |  |
| 9.3 | | Set the nitrogen mass flow (Channel 1) to 50% (500 cm3/min), using the Porter mass flow controller unit. |  |  |
| 9.4 | | Set the hydrogen mass flow (Channel 3) to 50% (250 cm3/min), using the Porter mass flow controller unit. |  |  |
| 9.5 | | Set the ethylene mass flow (Channel 4) to 50% (500 cm3/min), using the Porter mass flow controller unit. |  |  |
| 9.6 | | Close the nitrogen tank valve NTV1. |  |  |
| 9.7 | | Close the hydrogen tank valve HTV1. |  |  |
| 9.8 | | Close the ethylene tank valve ETV1. |  |  |
| 9.9 | | Watch the mass flow controllers to show 0.0% for the nitrogen, hydrogen and ethylene mass flows. |  |  |
| 9.10 | | Go to the wall with the gas tanks, opposite of the wall with the reactors. |  |  |
| 9.11 | | Check the pressure gauges on the wall opposite of the reactor, the gauges connected to the gas tanks. Check to make sure that NTP1, NTP2, HTP1, HTP2, ETP1, and ETP2 are all 0 psi. |  |  |
| 9.12 | | Close valves NTV2, point towards wall. |  |  |
| 9.13 | | Turn valve NTV4 to the right. |  |  |
| 9.14 | | Close valve HTV2, point towards wall. |  |  |
| 9.15 | | Turn valve HTV4 to the right. |  |  |
| 9.16 | | Close valve ETV2, point towards wall. |  |  |
| 9.17 | | Turn valve ETV4 to the right. |  |  |
| 9.18 | | Go back to the reactor and turn the mass flow controller knobs for nitrogen, hydrogen, and ethylene counterclockwise until they stop. |  |  |
| 9.19 | | Turn the valve NV to point upwards. |  |  |
| 9.20 | | Turn the valve HV to point upwards. |  |  |
| 9.21 | | Turn the valve EV to point upwards. |  |  |
| **10. - REMOVING SUBSTRATE FROM THE REACTOR VESSEL** | | |  |  |
| 10.1 | | Once the reactor vessel has cooled to room temperature disconnect the top and bottom of the reactor vessel from the reactor unit. | Be sure to grab the reactor vessel otherwise it will fall. | Use caution when handling glassware. |
| 10.2 | | Remove the reactor vessel from the heating unit. | Glass reactor vessel is made of glass and can break easily. Also, can be hot. | Handle with care. |
| 10.3 | | Remove the substrate from the reactor vessel. |  |  |
| **11. - CLEANING PROCEDURE OF THE REACTOR VESSEL** | | |  |  |
| 11.1 | | Place the reactor vessel back in the heating unit of the reactor. | Glassware use | Use caution when handling glassware. |
| 11.2 | | Connect the top of the reactor vessel to the reactor unit, this will send gases to the fume hood. |  |  |
| 11.3 | | Leave the bottom of the reactor open and place a fan underneath the reactor vessel. |  |  |
| 11.4 | | Set thermostat to ramp to 900°C in 90 minutes and then to hold at 900°C for 120 minutes. | Avoid burns, reactor oven set at 900°C | Wear insulating gloves or use caution when touching parts of the reactor. |
| 11.5 | | After heating the reactor vessel for the required amount of time, allow the reactor vessel to cool to room temperature. |  |  |
| 11.6 | | Remove the reactor vessel. |  |  |

