STANDARD OPERATING PROCEDURE-PALLADIUM ON CARBON (PD/C)

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| **CONTACT INFORMATION** | | | |
| **Location** | Building: | | Room: |
| **Street Address:** |  | | |
| **Lab Safety Contact:** | Name: | | |
| Lab Phone: | Office Phone: | |
| **Emergency Contact** | Name: | Phone: | |
| **TYPE OF STANDARD OPERATING PROCEDURE** | | | |
| Indicate which type of Standard Operating Procedure applies  Specific Process or Equipment  Specific Hazardous Chemical  Hazard Class for a Group of Chemicals | | | |
| **DESCRIBE PROCESS/EQUIPMENT, HAZARDOUS CHEMICAL or HAZARD CLASS** | | | |
| **Palladium on Carbon**  **Formula: Pd/C**  **CAS Number**: 7440-05-3  **Other names**: | | | |
| **HAZARD SUMMARY** | | | |
| Palladium on Carbon is a highly flammable finely divided metal. Palladium on Carbon is used as a hydrogenation  catalyst. Absorbed hydrogen during the process of hydrogenation makes it pyrophoric. It may ignite  spontaneously when dried in air. The use of flammable solvents and hydrogen gas during the process of  hydrogenation increases the risk of fire and explosion. | | | |
| SPECIAL HANDLING AND STORAGE REQUIREMENTS | | | |
| Dry solid materials may result in the formation of combustible dusts. The potential for combustible dust formation should be taken into consideration before additional processing occurs. Use in paste form (50-60% moisture) when the presence of water is not detrimental to the process.  Avoid heat, flames and sparks. The catalytic properties of this material will promote the oxidation and possible ignition of flammable liquids and vapors. The dehydrogenation of the lower alcohols (methanol) and compounds such as cyclohexane may readily cause ignition. After use, all catalysts which contain absorbed hydrogen may ignite when dried in air, especially in the presence of organic material.  Incompatible with alcohols, strong acids, bases, oxidizing agents. Palladium undergoes a violent reaction with arsenic. Contact with Hydrogen gas together with air will cause fire and explosion.  In case of fire use water spray, alcohol-resistant foam, dry chemical or carbon dioxide  Air and moisture sensitive.  Handle and store under inert gas.  Keep in a dry place.  Storage class (TRGS 510): 4.1B: Flammable solid hazardous materials | | | |
| **DESCRIBE PROCESS/EQUIPMENT, HAZARDOUS CHEMICAL or HAZARD CLASS** | | | |
| When setting up a hydrogenation reaction, the Pd/C should be added first and the vessel flushed with an inert gas. Then add the solvent. The inert gas should be introduced first, followed by the hydrogen gas. Or wet the Pd/C with water before adding the solvent.  If the hydrogenation reaction is run under pressure, use a blast shield.  If the reaction mixture is filtered through Celite or other filtration aid, do not let the filter cake dry out or it may ignite. Wash with water after solvent removal and do not allow to dry.  Hydrogenation using a balloon:  1) Evacuate the reaction vessel (this should have at least two openings) and backfill with an inert gas (nitrogen or argon).  2) Weigh out the desired amount of Pd/C and transfer into the reaction flask under an inert atmosphere.  3) Add a small amount of ethyl acetate, dichloromethane, or toluene to the reaction flask, making sure to wash down any Pd/C stuck to the flask walls. All the Pd/C should be submerged at this point.  4) Carefully add methanol by creating a stream down the side of the flask wall.  5) Add the reaction substrate either as a solution or neat.  6) Begin stirring the reaction mixture and then evacuate the flask just until the solvent begins to bubble, then carefully backfill with inert gas.  7) Repeat step 6 twice more.  8) Attach a balloon of hydrogen to your flask with an adapter that allows the balloon to be closed off from the reaction flask.  9) With the hydrogen balloon closed off, evacuate the flask until the solvent begins to bubble, and then open the balloon to the flask.  10) Repeat step 9 twice more.  Hydrogenation using a bomb reactor:  1) Purge the bomb with inert gas, and then follow steps 2-5 from the procedure above (“hydrogenation using a balloon”).  2) Seal the bomb under an inert atmosphere and then evacuate it using a mild vacuum source. Close the bomb off from the vacuum source and take it to the hydrogen source.  3) Connect the bomb directly to the hydrogen tank regulator using threaded fittings and fill the bomb with hydrogen up to the desired pressure (use the gauge on the bomb to determine the reaction pressure).  4) Close the bomb off from the hydrogen source so that it is now sealed. Close off the hydrogen cylinder using the regulator on the hydrogen tank, and disconnect your bomb from the regulator.  Workup:  1) For balloon reactions: Detach the hydrogen balloon from the flask and fill with inert atmosphere.  For bomb reactions: Connect a vent line to the bomb and then run the vent line to the back of the fume hood. Then slowly and carefully vent the reaction mixture to atmospheric pressure.  2) Filter the reaction mixture through a bed of Celite (or similar filter aid)  3) Taking care not to let the filter cake filter to dryness, wash with the desired solvent (typically the same solvent used in the reaction)  4) Disconnect the filter from the receiving flask, and then add several mL of water to the filter.  5) Discard the slurried Pd/C and filter aid in a dedicated waste jar that contains water.  NOTE: If you must add catalyst to solution, prepare a slurry of Pd/C in dichloromethane or toluene (using steps 1-3 above) and then add this slurry to your reaction mixture. However, this should not be seen as a preferred method. | | | |
| **ENGINEERING AND VENTILATION CONTROLS** | | | |
| Locate the nearest fire extinguisher before beginning work. Work under an inert atmosphere (argon, nitrogen), in a glove box, manifold, or an enclosed inert environment within a fume hood.  Provide appropriate exhaust ventilation at places where dust is formed.  Reactions under pressure should be placed behind a blast shield unless they are in a bomb reactor rated to withstand the reaction pressure. | | | |
| **PERSONAL PROTECTIVE EQUIPMENT** | | | |
| **PPE Requirements:**  Long pants or clothing that covers all skin below the waist  Shoes that cover the entire foot  Gloves; indicate type: Nitrile or latex (must be clean and dry)  Inspect gloves before use. Use proper glove removal technique to avoid skin contact with outer surface of glove. Wash hands after removing gloves.  Safety goggles  Safety glasses  Face shield  Lab coat  Flame-resistant lab coat  Other:  If the use of an N95, half mask, or full face respirator is requested, the individual and/or their supervisor must first contact Environmental Health & Safety for a consultation to determine if respirator use is necessary. If EH&S determines the use of a respirator is necessary, the individual must participate in the University’s respirator program. This includes a medical evaluation; respirator fit test, and training. | | | |
| **EMERGENCY PROCEDURES** | | | |
| In case of fire or large and/or extremely hazardous chemical releases pull the fire alarm and evacuate the area  If someone is seriously injured or unconscious  **CALL 911 or CAMPUS POLICE AT <enter your campus PD #>**  From a safe place, provide as much information as possible to the emergency responders including chemical name, volume, hazards, injuries, and location.  **Chemical Exposure**: Remove any contaminated clothing, and IMMEDIATELY flush contaminated skin with water for at least 15 minutes following any skin contact. For eye exposures, IMMEDIATELY flush eyes with water for at least 15 minutes. Consult SDS for guidance on appropriate first aid. Where medical attention is required, bring the SDS(s) of chemical(s) to aid medical staff in proper diagnosis and treatment.  **Evacuation Procedure**   * Immediately evacuate the building via the nearest exit when the fire alarm is activated. * If unable to evacuate due to a disability, shelter in the area of rescue / refuge, typically a stairwell landing, and wait for assistance from drill volunteers or emergency responders. * Instruct visitors and students to evacuate and assist them in locating the nearest exit. * Do not use elevators to exit the building during an evacuation as they may become inoperable. * Carry only those personal belongings that are within the immediate vicinity. * Close doors to limit the potential spread of smoke and fire. * Terminate all hazardous operations and power off equipment. * Close all hazardous materials containers. * Remain outside of the building until the building is released for reentry. * Do not restrict or impede the evacuation. * Convene in the designated grassy gathering area and await instruction from emergency responders or drill volunteers. Avoid parking lots. * Report fire alarm deficiencies, (e.g., trouble hearing the alarm) to facilities personnel for repair. * Notify evacuation drill volunteers or emergency responders of persons sheltering in the areas of rescue/ refuge. * **Never assume that an alarm is a “false alarm”. Treat all fire alarm activations as emergencies. Get out of the building!**   **Incident and Near Miss Reporting**: Report any incident that occurs in any University of South Florida affiliated teaching or research laboratory/studio or field research project. An incident means any unplanned event within the scope of a procedure that causes, or has the potential to cause, an injury or illness and/or damage to equipment, buildings, or the natural environment. Due to medical privacy concerns, no personal medical information of the person involved in the incident shall be entered on or submitted with the form.  <http://www.usf.edu/administrative-services/environmental-health-safety/reporting/index.aspx>  **Workers’ Compensation Procedure:** Supervisor and employee (if possible) call AmeriSys at 800-455-2079 to report a work-related injury or illness. Complete the [Consolidated Injury/Illness Reporting Form](https://www.usf.edu/administrative-services/environmental-health-safety/reporting/injury-illness-reporting.aspx), and send it to EH&S within 24 hours. | | | |
| **WASTE DISPOSAL** | | | |
| All used, filtered catalysts should be kept wet and out of contact with combustible liquids and vapors. Palladium waste should be separated from the mixture and placed in a labeled container that contains ten times the volume of water. Any filtration aids such as Celite should also be discarded in the palladium waste stream. It is important to make sure any solid waste from a hydrogenation has been exposed to water prior to disposal.  All chemical waste generated within USF System laboratories is considered hazardous waste and must be disposed of as hazardous waste in accordance with the USF Hazardous Waste Management Procedure, the U.S. EPA, and the FDEP. The USF Hazardous Waste Management Procedure can be found using the following link, <https://www.usf.edu/administrative-services/environmental-health-safety/documents/hazwaste-managementprocedure.pdf> | | | |
| **TRAINING REQUIREMENTS** | | | |
| All individuals working with chemicals in USF laboratories must take EH&S’s Laboratory Safety Training. To register for Laboratory Safety Training, please use the following link, <https://www.usf.edu/administrative-services/environmental-health-safety/training/course-descriptions.aspx#labsafety>  This procedure may warrant additional safety training per the PI, EH&S, or an authorizing unit such as the Biosafety or Radiation Safety programs. Check training requirements for this activity below:  Research Specific Training from the PI/Lab Supervisor or their designee  EH&S Laboratory Safety Training  EH&S Hazard Communication  EH&S Hazardous Waste Awareness and Handling  EH&S Respirator Fit Test  EH&S Biomedical Waste  EH&S Hazardous Waste Pharmaceutical Training  EH&S Fire Prevention Safety  EH&S Slips, Trips, and Falls  RIC Biosafety Core Course  RIC Shipping Biohazardous Materials  RIC BSL 3  RIC Radiation Safety  RIC Laser Safety  RIC Boating Safety  RIC Scientific Diving  Other:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ | | | |
| **PRIOR APPROVALS** | | | |
| This activity requires prior approval from the PI/designee.  If this box is checked, working alone is not allowed. | | | |

By signing and dating here the Principal Investigator/ or a designee certifies that the Standard Operating Procedure (SOP) for ***Palladium on Carbon*** is accurate and effectively provides safe standard operating procedures for employees and students in this lab who will handle this hazardous chemical.

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Signature Printed Name Date

I affirm that I have read and understand the Standard Operating Procedure for ***Palladium on Carbon*** and have undergone the EH&S Laboratory & Research training and any lab specific training regarding this SOP.

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| Printed Name | Signature | Date |
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