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| **Brueckner Lab-Specific Standard Operating Procedure (LSOP):**  **Hydrogenation Metal Catalysts** | |
| **Principal Investigator(PI):** Christian Brueckner | |
| **Building:** Chemistry | **Lab(s) Covered by LSOP:** R413/R415 |
| **Department:** Chemistry | **Lab Phone Number(s):** 6-6596/6-6598 |
| **SECTION 1 – HAZARDOUS CHEMICAL(S) or PROCESS(ES) and HAZARDS INVOLVED** | |
| **Raney Ni, Pd/C:** These catalysts are pyrophoric materials and it ignites spontaneously when exposed to air. They also tend to be associated with flammable solvents. Also include common hazards such as corrositivity, water reactivity, peroxide formation, and toxicity. It can prove fatal if ingested or inhaled. Exposure can cause serious damage to lungs and skin. Nickel can be a potent allergen. Target Organs: Skin, Kidney, Liver, and Lungs. Known carcinogens. | |
| **SECTION 2 – ADMINISTRATIVE CONTROLS** | |
| * Anyone using the chemicals and procedures described herein needs to have undergone the annual EH&S [Chemical Hygiene Training](http://www.ehs.uconn.edu/Chemical/?p=training) * Be aware of the applicable safety data sheets (SDS): <http://www.msds.com> (Raney Ni-[SDS](https://www.fishersci.com/shop/msdsproxy?storeId=10652&productName=AC395925000), Pd/C-[SDS](http://www.cen.iitb.ac.in/chemical_approval/msds/314_msds.pdf)). * [Working Alone](http://policy.uconn.edu/2012/07/30/working-alone-policy/) is not permitted when using chemicals or processes described in this LSOP. * Any reaction flasks containing hydrogenation catalysts/hydrogen must be clearly labelled as such with an appropriate warning sings (e.g, DANGER- Raney Nickel). * Hydrogenations should be performed during normal business hours (i.e., 8:00 am-5:00 pm Mo-Fri), if possible. * Anyone using the chemicals and procedures described herein need to be aware of the applicable SDS. * [Working Alone](http://policy.uconn.edu/2012/07/30/working-alone-policy/) is not permitted when using chemicals or processes described in the LSOP. * Keep away from sources of ignition and combustible materials and remove any excess clutter and any flammable solvents that are not needed for the reaction. | |
| **SECTION 3- ENGINEERING CONTROLS** | |
| * All research involving hydrogenation catalysts must be conducted in a chemical fume hood, under dry conditions, with the sash at the lowest working height and with sliding sash panels (if applicable) aligned to form a barrier between the researcher and the experiment. * Chemical fume hoods must have been tested by EHS within the last year. If the hood is not working properly, contact Facilities (486-3113) to repair the hood or EH&S to retest (486-3613). | |
| **SECTION 4 – WORK PRACTICES** | |
| * Be aware of catalysts dusts – that might be pyrophoric, especially when in contact with hydrogen gas * Design a quenching scheme for residual materials prior to using materials. * Design your experiment to use the least amount of material possible to achieve the desired result. * It is better to do multiple transfers of small volumes than attempt to handle larger quantities. Before transferring, make sure that the material is at room temperature. * Verify your experimental set-up and procedure prior to use. * Label the reaction flask with a sign saying "Pyrophorics In-Use Area". * Ensure that the area is properly equipped with a properly functioning eye wash/safety shower within ten seconds of travel. * At the end of each experiment, thoroughly inspect the area for residual reactive material. | |
| **SECTION 5 – PERSONAL PROTECTIVE EQUIPMENT (PPE)** | |
| * At a minimum, a lab coat, long pants as well as closed-toed footwear and chemical safety glasses that meet American National Standards Institute (ANSI) standard Z-87.1 must be worn when handling OsO4 * Nitrile Gloves must be worn while handling the hydrogenation catalysts | |
| **SECTION 6 – STORAGE** | |
| * Pd/C Store in a dry place (locked filing cabinet with the precious metal catalysts in R415). Keep cool and protect from sunlight and humid air/moisture. Whenever possible, store in a desiccator. Otherwise, store inside a container with an inert atmosphere (Nitrogen, Argon etc.). * Raney-Ni comes as an aqueous slurry that is either kept in the inorganics shelf in R415 or in the fridge * Keep container tightly closed when not in use. * Minimize dust generation and accumulation. * Container must be labeled with the chemical name and hazard communication label contain hazard classes. | |
| **SECTION 7 – SPILL AND ACCIDENT PROCEDURES** | |
| * When either of the catalysts is spilled, cover with sand or vermiculite and sweep up, avoiding the generation of dusts * Dispose of in the solid waste cans * If a small fire occurs, do not panic, but simply cover the flask or funnel in which there is a fire with a watch glass and it will go out. Have a suitable sized watch glass on hand.   **Report any incident to the PI and fill out the** [**accident form**](https://www.google.com/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&ved=0ahUKEwiF3bPe1dPXAhVRRN8KHX4wDf4QFggmMAA&url=https%3A%2F%2Fchemistry.uconn.edu%2Fwp-content%2Fuploads%2Fsites%2F1259%2F2015%2F09%2FIncident-Report-Form.doc&usg=AOvVaw3Uov8IQ2Z-Kan) | |
| **SECTION 8 – FIRST AID PROCEDURES** | |
| *Eyes*   * Immediately move to the eyewash station, hold eyelids open and flush with water. Remove contact lenses while flushing (if applicable) * Have another person from the lab dial **911** and specifically mention specific exposure * Continue flushing the eyes until emergency personnel arrive   *Skin*   * Immediately move to safety shower or other water source and begin rinsing affected area(s). Remove contaminated clothing (if applicable) while flushing. * Have another person from the lab dial **911** if intense skin irritation is observed and specifically mention specific exposure * Keep rinsing affected area(s) until emergency personnel arrives   *Ingestion*   * Immediately rinse the mouth with cold water * Do NOT induce vomiting * Have another person from the lab dial **911** and specifically mention specificexposure   *Inhalation*   * Move to fresh air * Dial **911** and inform emergency responders that the accident involved the specific reagent   **Report any incident to the PI and fill out the** [**accident form**](https://www.google.com/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&ved=0ahUKEwiF3bPe1dPXAhVRRN8KHX4wDf4QFggmMAA&url=https%3A%2F%2Fchemistry.uconn.edu%2Fwp-content%2Fuploads%2Fsites%2F1259%2F2015%2F09%2FIncident-Report-Form.doc&usg=AOvVaw3Uov8IQ2Z-Kan) | |

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| **SECTION 9 – WASTE MANAGEMENT** |
| * All waste must be labeled with “Hazardous Waste” stickers or tags, use full chemical names to describe the waste (i.e., no chemical abbreviations or symbols), be stored in sturdy containers with tight-fitting caps or lids, and be stored alone or with other compatible chemicals. * The solid catalysts get disposed of in the solid waste cans. Especially Raney-Ni, if dried out, tends to catch fire. Mix with sand before disposal. Leave sand/Ni mix in fume hood for a day before disposal. * Hazardous wastes must be stored at or near a green “Satellite Accumulation Area” sign prior to disposal by EHS. Once the containers are 80% filled, fill our EH&S chemical [waste pickup form](http://ehs.uconn.edu/Regulated%20Waste%20Management/index.php). * The [Chemical Waste Disposal Manual](http://ehs.uconn.edu/Chemical/ChemWasteDisp.pdf) must be used as a reference. |
| **SECTION 10 – DECONTAMINATION PROCEDURES** |
| * Equipment can be decontaminated through rinse with copious amounts of water; use of surfactants is recommended. * Wash your hands after handling any of the hydrogenation catalysts |
| **SECTION 11 – SPECIFIC PROCEDURE** |
| A specific procedure has been described in following work: Sharma, M.; Banerjee, S.; Zeller, M.; Brückner, C. ‘Fusion and Desulfurization Reactions of Thiomorpholinochlorin’ *J. Org. Chem.* **2016**, *81*, 12350−12356.  To a solution of [*meso*-tetraphenyl-2,3-dimethylene-2a-thio-2a-homoporphyrinato]nickel(II) **8Ni** (25 mg, 3.43 × 10−5 mol) in THF (10 mL) was added Raney-Ni (2.0 mL of a 50% slurry of Raney-Ni in H2O), and the reaction mixture was stirred for 12 h at rt under a H2 atmosphere (provided by balloon). After the starting material was consumed (reaction monitored by TLC), the catalyst was filtered off by gravity filtration. The organic phase was dried over anhydrous Na2SO4, filtered, and evaporated to dryness by rotary evaporation. |

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| **SECTION 12A. APPROVAL** | | | |
| I have reviewed, understand and agree to follow this lab-specific standard operating procedure (LSOP) involving hydrogenation catalysts*.* Failure to follow this LSOP or lab-specific training guidelines is a violation of the [*University Health & Safety Policy*](http://policy.uconn.edu/2011/05/19/health-and-safety-policy/) and [*University Code of Conduct*](http://policy.uconn.edu/2011/05/17/employee-code-of-conduct/).  Further approval and/or review of this LSOP by the PI/Supervisor is required if any of the following events occur:   * A significant change in amount (i.e., doubling of the scale of reaction) or substitution of the chemicals in the procedure is planned * A major change in the agreed-upon experimental set-up is planned (heating instead of room temp, etc.) * Any signs of a failure in safety design or equipment are observed * Any signs or symptoms of a chemical exposure to any personnel are observed * Unexpected and/or potentially dangerous experimental results occur (e.g., fire, uncontrolled buildup of heat and/or pressure, etc.) | | | |
| **Researcher Name/Signature** | **Trainer Name/Signature** | **Training Date** | |
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| **SECTION 12B. PRINCIPAL INVESTIGATOR CERTIFICATION** | | | |
| I approve the contents of the lab-specific standard operating procedure listed above. | | | |
| **PI Signature:** | | | **Date:** |
| **A HARD OR ELECTRONIC COPY (https://bruckner.research.uconn.edu/safety-resources/) OF EACH LAB-SPECIFIC STANDARD OPERATING PROCEDURE MUST BE READILY AVAILBALE IN THE LAB.** | | | |