|  |  |
| --- | --- |
| **Brueckner Lab-Specific Standard Operating Procedure (LSOP):**  **Strong Aqueous, Non-oxidizing Mineral Acids: HCl, HBr, HI, H2SO4, H3PO4, HClO4** | |
| **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** | |
| All compounds in this class are highly corrosive chemicals. Inhalation may cause irritation to the respiratory tract with burning pain in the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema. Contact with skin causes burns and irritation. Eye contact causes burns, irritation, and may cause blindness. Ingestion may cause permanent damage to the digestive tract.  Additional notes on chemical-specific hazards:  *Hydrochloric (HCl), hydrobromic (HBr), and hydroiodic (HI) acids*: strong eye irritants and lachrymators  *Hydrofluoric acid (HF)*: causes severe burns that may not be immediately painful or visible – symptoms may be delayed 8 hours or longer – the use of HF is **not** permitted in the Brueckner Laboratories  *Sulfuric acid (H2SO4)* – water reactive; strongly dehydrating, very corrosive toward tissue  *Phosphoric acid (H3PO4)* –toxicity towards a growing embryo or the reproductive systems | |
| **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 (MSDS): <http://www.msds.com> * [Working Alone](http://policy.uconn.edu/2012/07/30/working-alone-policy/) is not permitted when using chemicals or processes described in this LSOP * Handling of the large bottles of the concentrated acids should be performed during normal business hours (i.e., 8:00 am-5:00 pm Mon-Fri), if possible * Unobstructed eyewash and safety shower must be in the immediate work area | |
| **SECTION 3- ENGINEERING CONTROLS** | |
| All research with the volatile conc. acids (HCl, HBr, HI) must be conducted in a chemical fume hood, 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** | |
| All containers of strong acids must be clearly labeled with the chemical name and hazard classes and kept tightly-sealedDo not allow water to get into the containers of all acids but particularly H2SO4 because of the strongly exothermic heat of dilutionUse only with adequate ventilationDo not breathe spray or mistDo not use with metal spatula or other metal itemsWhen diluting acids, always add acid (slowly) to cooled water **(Never add water to acid)**Strong acids may react with some metals to produce hydrogen gas, which is flammable/explosive | |
| **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 acids * Well-fitted chemical splash goggles or a full-face shield are required when handling larger quantities (>500 mL) of the conc. acids * Nitrile Gloves must be worn while handling small quantities of the acids * Heavier, long-sleeved gloves and an apron are recommended when handling larger quantities of conc. (>500 mL) acids. | |
| **SECTION 6 – STORAGE** | |
| * Store acids as indicated in safety data sheets (SDSs): <http://www.msds.com/> * Ensure labels on original bottles remain legible and prominently displayed to identify contents * Ensure both original and secondary containers remain intact and are stored with tight-fitting caps or lids * Keep container closed when not in use * The large (>250 mL) bottles of strong acids should be stored in isolation from all other chemicals in an approved acid or corrosives safety cabinet lined with a plastic secondary tray: acid cabinet in R415 * Do not store near alkaline substances * Strong acids are incompatible with metals, oxidizing agents, reducing agents, bases, acrylonitrile, chlorates, finely powdered metals, nitrates, perchlorates, permanganates, carbides, fulminates, picrates, organic materials. This list is representative and may not be comprehensive | |
| **SECTION 7 – SPILL AND ACCIDENT PROCEDURES** | |
| * If any large (>250 mL) of a conc. acid occurs outside of the fume hood, warn the other occupants in the lab and evacuate the laboratory immediately if the acid is volatile * Close door(s) to lab and post a “**NO ENTRY**” sign(s) explicitly mentioning the type of hazard * Activate the fire alarm and call **911** in case of a large (>1000 mL) spill outside the fume hood * Do not re-enter area until instructed to do so by an emergency personnel   **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 acid 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) with copious amounts of water. Dilutions is the solution!!! Remove contaminated clothing (if applicable) while flushing; safety showers are also in the washrooms but do not use alone in an emergency. * Have another person from the lab dial **911** if intense skin irritation is observed and specifically mention specific acid exposure * You may use dil. aqueous NaHCO3 solution to affected area(s) until emergency personnel arrive   *Ingestion*   * Immediately rinse the mouth with cold water * Do NOT induce vomiting * Have another person from the lab dial **911** and specifically mention the specific acid involved   *Inhalation*   * Move to fresh air * Dial **911** and inform emergency responders that the accident involved the specific acid * **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 9 – WASTE MANAGEMENT** | |
| * All acid wastes 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 * 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** | |
| **Work Area**   * Decontaminate equipment and bench tops using soap and water; a dil. aqueous sodium bicarbonate solution may be used to neutralize the surfaces; check with pH paper * Dispose of the spent acids and disposables contaminated with acids as hazardous waste   **Personal Hygiene**   * Storage/Consumption of food or beverage is prohibited in all laboratory areas * Use standard chemical hygiene practices regarding PPE (see above). * Wash hands thoroughly after handling acids * Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with the acid | |
| **SECTION 11 – SPECIFIC PROCEDURE** | |
| |  | | --- | | A typical use of sulfuric acid is described in: Yonezawa, T.; Shafie S. A.; Hiroto S.; Shinokubo H. ‘Shaping Antiaromatic -Systems by Metalation: Synthesis of a Bowl-Shaped Antiaromatic Palladium Norcorrole’, *Angew. Chem.* *Int. Ed.* **2017**, *56*, 11822–11825.  CAUTION: Fumehood and eye protection required!  *Free-Base Dimesitylnorcorrole* **(2H2):**  A round-bottomed flask containing bis(diiododipyrrin) copper complex **2Cu** (27.2 mg, 25.0 *μ*mol) and copper(I) 2-thiophenecarboxylate (143 mg, 750 *μ*mol) was filled with N2 and dry NMP (15 mL) was added. The solution was stirred at room temperature for 20 min. The resulting mixture was extracted with AcOEt. The combined organic layers were washed with water and brine, and dried over Na2SO4. After removing the solvent by rotary evaporation, the round-bottomed flask containing the residue was filled with N2 and dry CHCl3 (2.6 mL) was added. The solution was stirred at –65 °C for 10 min. To this solution, H2SO4 (1 mL) was added carefully under stirring condition and the stirring was continued for 5 min. The reaction mixture was quenched with aqueous NaHCO3 solution and aqueous layer was washed by CHCl3. The combined organic layers were dried by brine and Na2SO4. After removing the solvent by rotary evaporation, the residue was purified by column chromatography (hexane CHCl3 as eluent) to afford free-base dimesitylnorcorrole **2H2** (6.83 mg, 13.1 *μ*mol, 52%). | | |

|  |  |  |
| --- | --- | --- |
| **SECTION 12A. APPROVAL** | | |
| I have reviewed, understand and agree to follow this lab-specific standard operating procedure (LSOP)*.* 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** |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
| **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.** | | |