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| **Brueckner Lab-Specific Standard Operating Procedure (LSOP):**  **Boron Trifluoride Etherate (BF3·OEt2)** | | | | | |
| **Principal Investigator(PI):** Christian Brueckner | | | | | |
| **Building:** Chemistry | | | | | **Lab(s) Covered by LSOP:** R413/R415 |
| **Department:** Chemistry | | | | | **Lab Phone Number(s):** 6-6596/6-6598 |
| **Boron trifluoride (BF3)** | |  | | **Acute toxicity:**  adverse effects occurring following oral or dermal administration of a single dose of a substance, or multiple doses given within 24 hours, or an inhalation exposure of 4 hours.  **Skin corrosion**: is the production of irreversible damage to the skin; namely, visible necrosis through the epidermis and into the dermis  ***Irritant*:** refers to a substance that can cause temporary inflammation of living tissue at the point of contact. | |
| **SECTION 1 – HAZARDOUS CHEMICAL(S) or PROCESS(ES) and HAZARDS INVOLVED** | | | | | |
| Boron trifluoride (BF3·OEt2) is corrosive and can be toxic if ingested. Inhalation of BF3·OEt2 is hazardous and may cause irritation of the nose, throat, and lungs, leading to coughing and shortness of breath. BF3 is highly flammable and corrosive and reacts violently with water or moist air. It is incompatible with strong oxidizing agents, acids, bases, alcohols, and alkali metals. BF3·OEt2 may also cause chronic digestive tract irritation. Repeated exposure may cause dryness of the nose and nosebleeds. More serious effects include kidney and neurological damage. High exposure to BF3 can cause fluorosis, causing pain, disability, and mottling of the teeth. Carcinogenic and teratogenic effects: N/A | | | | | |
| **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> * [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 50+ mL quantities of the acid should be performed during normal business hours, if possible * An eyewash and safety shower must be in the immediate work area of boron trifluoride use | | | | | |
| **SECTION 3- ENGINEERING CONTROLS** | | | | | |
| All research with boron trifluoride 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 | | | | | |
| **SECTION 4 – WORK PRACTICES** | | | | | |
| Ensuring chemical processes for BF3·OEt2 are in enclosed spaces to prevent corrosion and/or irritationAll containers of BF3·OEt2 must be clearly labeled with the chemical name and hazard classes and kept tightly-sealedAll work with BF3·OEt2 must be performed on a chemically-compatible secondary containment trayEmpty containers of BF3·OEt2 must be handled carefully since product residues (vapors, liquid) are still harmfulUse local exhaust to avoid exposure | | | | | |
| **SECTION 5 – PERSONAL PROTECTIVE EQUIPMENT (PPE)** | | | | | |
| * At a minimum, chemical splash goggles or safety glasses that meet American National Standards Institute (ANSI) standard Z-87.1 must be worn when handling BF3·OEt2 * PIs must determine when or if full-face shields are required when working with BF3·OEt2 * Nitrile Gloves must be worn while handling small quantities of BF3·OEt2 * A properly fitting lab coat must be worn * Long pants, as well as, closed-toed footwear (which covers the entire foot) must be worn when working | | | | | |
| **SECTION 6 – STORAGE** | | | | | |
| * Store BF3 away from oxidizing agents, strong acids or bases, and alkali metals, and water as indicated in safety data sheets (SDSs) <http://www.ehs.uconn.edu/information/> * 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 * Stored away from strong oxidizing agents, acids, bases, alcohols, and alkali metals | | | | | |
| **SECTION 7 – SPILL AND ACCIDENT PROCEDURES** | | | | | |
| * Evacuate the laboratory immediately * 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 (>500 mg) spill outside the fume hood * Do not re-enter area until instructed to do so by UCFD or other 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 BF3·OEt2 exposure * Continue flushing the eyes until emergency personnel arrives   *Skin*   * Immediately move to safety shower or other water source and begin rinsing affected area(s). Remove contaminated clothing (if applicable) while flushing * You may use a dilute aqueous bicarbonate solution to rinse the affected areas * Have another person from the lab dial 911 and specifically mention BF3·OEt2 exposure * Keep rinsing affected area(s) until emergency personnel arrives   *Ingestion*   * Immediately rinse the mouth with cold water * Do NOT induce vomiting * Do NOT give emetics or baking soda * Have another person from the lab dial 911 and specifically mention BF3·OEt2 exposure * If the victim is conscious, have them drink several glasses of milk or several ounces of milk of magnesia, Mylanta®, Maalox®, etc. * If milk or antacids are not available continue drinking water until emergency personnel arrives   *Inhalation*   * Move to fresh air * Dial 911 and specifically mention BF3·OEt2 exposure, including to the first responders   **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 BF3·etherate 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, glass 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 | | | * Equipment can be decontaminated through rinse with water and a surfactant or an aqueous bicarbonate solution | | |
| Personal Hygiene | | | * Wash hands thoroughly after handling BF3·OEt2 | | |
| **SECTION 11 – SPECIFIC PROCEDURE** | | | | | |
| A typical use of BF3·etherate in our laboratories is described in: Lindsey, J. S.; Schreiman, I. C.; Hsu, H. C.; Kearney, P. C.; Marguerettaz, A. M. ‘Rothemund and Adler-Longo reactions revisited: synthesis of tetraphenylporphyrins under equilibrium conditions’ *J. Org. Chem.* **1987,** *52*, 827-36.  A 2-L, three-necked, round-bottomed flask fitted with a reflux condenser and nitrogen inlet port was filled with 1 L of distilled CH2Cl2. Samples of benzaldehyde (1.525 mL, 0.015 mol, 1.5 x 10-2 M) and pyrrole (1.04 mL, 0.015 mol, 1.5 x 10-2 M) were added and the solution was stirred magnetically at room temperature under a slow steady stream of nitrogen. After 15 min, BF3·etherate (0.4 mL of a 2.5 M solution in CH2Cl2, 10-3 M) was added and the reaction vessel was shielded from ambient lighting. After 1 h, p-chloranil (2.77 g, 0.011 mol, 3 equiv per porphyrinogen) in powder form was added all at once to the reaction vessel. The flask was immersed in a water bath preheated to 45 °C and the solution was refluxed for 1 h. The solution was then concentrated to about 50 mL by rotary evaporation, and 10-12 g of Florisil was added. The slurry was further dried to afford a damp dark powder, which was poured onto the top of a chromatography column (2.5-cm diameter) filled with Florisil (38 cm in height). The column was washed with about 500 mL of CH2Cl2/petroleum ether (3:1) to elute several small bands of fast moving pigments, followed by 300-400 mL of CH2Cl2 to elute the porphyrin. The first two porphyrin fractions contained 10% impurities while the third and final fraction appeared pure. These three porphyrin fractions were combined and concentrated to afford a crude product weighing 1.254 g (54%), which upon recrystallization gave 1.033 g of tetraphenylporphyrin (45% yield overall). | | | | |

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| **SECTION 12A. APPROVAL** | | |
| I have reviewed, understand and agree to follow this lab-specific standard operating procedure (LSOP) for BF3·OEt2*.* 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.** | | | |