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| **Brueckner Lab-Specific Standard Operating Procedure (LSOP):**  **Concentrated Nitric Acid (HNO3)** | |
| **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** | |
| Concentrated nitric acid (also known as *aqua fortis*) is an extremely corrosive acid and strong oxidizing agent. It may be harmful if ingested, inhaled, or absorbed through the skin. It can cause severe skin and eye burns resulting in irreversible damage. It is extremely destructive to the tissue of the mucous membranes and the upper respiratory tract. The main use of nitric acid is in the production of agricultural fertilizers. Its other uses include the production of nylon precursors, explosives, and rocket fuel. Nitric acid is an oxidizer that may intensify fires. Fire conditions may cause formation of hazardous nitrogen oxides. Nitric acid may be harmful if inhaled, ingested, or absorbed through the skin. It is extremely destructive to the tissue of the mucous membranes and upper respiratory tract. Causes severe skin and eye burns. May cause blindness and permanent eye damage. Inhalation may cause spasms, inflammation and edema of the bronchi or larynx, and pneumonitis. Other symptoms include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, vomiting, and pulmonary edema. Effects may be delayed. Large doses may cause conversion of hemoglobin to methemoglobin, producing cyanosis or a drastic fall in blood pressure, leading to collapse, coma, and possibly death. Chronic exposure may cause erosion of the teeth, jaw necrosis, and kidney damage. | |
| **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 * Experiments should be performed during normal business hours (i.e., 9:00 am-5:00 pm Mon-Fri), if possible * An eyewash and safety shower must be in the immediate work area and not obstructed where strong acids are being used | |
| **SECTION 3- ENGINEERING CONTROLS** | |
| All research involving conc. HNO3 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 experimentChemical 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 EHS 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-sealedAll work with acids must be performed on a chemically-compatible secondary containment trayDo not allow water to get into the container because of violent reactionUse 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 and potentially explosiveConc. HNO3 is also a strong oxidizer that may react violently with organic compounds | |
| **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 strong acids * A full-face shield and/or a rubber apron is recommended when working with large quantities (>100 ml) of conc. HNO3 * Handle with gloves: Viton gloves with long sleeves are recommended when handling larger quantities. * Nitrile gloves are not recommended for concentrated (>70%) nitric acid according to the Ansell Chemical Resistance Guide * Gloves must be inspected prior to use * Use proper glove-removal technique (without touching glove’s outer surface) to avoid skin contact with this material * 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** | |
| * Nitric acid should be stored in a secondary container inside a designated corrosive cabinet * Nitric acid should be stored in a separate area from any bases due to the danger of an exothermic reaction if allowed to mix * As an oxidizing agent, nitric acid should be kept separate from organics that are flammable | |
| **SECTION 7 – SPILL AND ACCIDENT PROCEDURES** | |
| * Spill – Assess the extent of danger * Help contaminated or injured persons * Evacuate the laboratory immediately if larger spills involving >250 mL conc. HNO3 are involved - avoid breathing vapors * Close door(s) to lab and post a “**NO ENTRY**” sign(s) explicitly mentioning the type of hazard * If safe, you may cover the spill with sand or vermiculite or another inert absorbent – never use paper * Activate the fire alarm and call **911** in case of a large (>500 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 HNO3 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 * Have another person from the lab dial **911** if intense skin irritation is observed and specifically mention HNO3 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 HNO3 exposure   *Inhalation*   * Move to fresh air * Dial **911** and inform emergency responders that the accident involved HNO3   **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 strong 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 plastic or 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**   * Decontaminate equipment and bench tops using soap and copious amounts of water   **Personal Hygiene**   * Use standard chemical hygiene practices regarding PPE (see above). * Wash Viton gloves after handling nitric acid * Wash hand thoroughly after handling nitric acid | |
| **SECTION 11 – SPECIFIC PROCEDURE** | |
| A typical use of nitric acid is described in: Ostrowski S., Szerszeń D., Ryszczuk M. ‘Electrophilic Nitration of *meso*-Tetraarylporphyrin Complexes at the Pyrrolic Position’, *Synthesis* **2005**, 5, 819–823.  CAUTION: Fumehood and eye protection required!  ***Nitration of Metallo-5,10,15,20-tetraarylporphyrins; General Procedure***  *meso*-Tetraarylporphyrin complex (**1a/1b**; 0.10 mmol) was dissolved in dry CHCl3 (140 mL) and the solution was stirred under argon at r.t. in a light-shielded flask. To this mixture, a 25% aq. solution of nitric acid (24 mL, 103 mmol) was added dropwise via syringe. The reaction was continued for 0.5 h (with TLC monitoring). Then, it was washed with water to reach pH c 7 (6 × 50 mL). The water phases were extracted once with CHCl3 (70 mL), and the combined organic layers were dried with MgSO4/Na2CO3. After evaporating the solvent, the crude residue was separated on a chromatographic column, using a mixture of CHCl3–*n*-hexane as the eluent (2:1) to give: a) From compound **1a**: Compound **2a** (58 mg, 81%) and the mixture of dinitrated products (12.0 mg, yield 16%), from which **3a** (4.6 mg, 6%) and **4a** (2.6 mg, 3%) were isolated and characterized. | |

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