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| **Brueckner Lab-Specific Standard Operating Procedure (LSOP):**  **Osmium Tetraoxide (OsO4) either as pure solid or in solution in pyridine or water** | | |
| **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** | | |
| The acute toxicity of the light-yellow crystals of osmium tetraoxide (OsO4) is high. It can cause severe damage to the cornea of the eye. Because it is very volatile, extreme care must be taken when working with it. In low concentrations, irritation and lacrimation can occur; the appearance of rings around lights can be observed. Upon heavier exposure, irreversible eye damage can occur. Symptoms may not be noted until several hours after exposure. Contact with OsO4 can cause headaches, coughing, dizziness, lung damage, and may be fatal. Carcinogenic and reproductive effects: N/A. Target Organs: Eyes, Liver, Kidney | | |
| **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 * Any reaction flasks containing OsO4 need to be clearly labelled as such * Osmylations should be performed during normal business hours (i.e., 8:00 am-5:00 pm Mon-Fri), especially when they involve larger than 500 mg of OsO4 * An eyewash and safety shower must be in the immediate work area where OsO4 is used * A bottle of corn oil should be in close vicinity when working with OsO4 | | |
| **SECTION 3 – ENGINEERING CONTROLS** | | |
| All research with OsO4 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 for a retest (486-3613) | | |
| **SECTION 4 – WORK PRACTICES** | | |
| OsO4 must be handled and stored in a dry place while it’s kept cool and protected from sunlight.All containers must be clearly labeled with the chemical name and hazard classes and kept tightly-sealedEmpty containers of OsO4 must be handled carefully since product residues may still be harmful: Leave all contaminated glassware in the fume hood for at least a dayBe aware that the rotary evaporator used to evaporate solvent used in an osmylation reaction may still contain OsO4 – rinse carefully right after use. | | |
| **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 * Chemical splash goggles are required when dividing up an ampule of OsO4 * Nitrile Gloves must be worn while handling small quantities of OsO4 * When opening an ampule, leather work gloves worn over the nitrile gloves when an ampule of OsO4 is broken open | | |
| **SECTION 6 – STORAGE** | | |
| * Store OsO4 as indicated in safety data sheets (SDS): <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 * Store opened ampules only for minutes under a fume hood. Dissolve excess OsO4 in a volumetric flask in pyridine and store the deep-yellow solution stoppered under fume hood for a few days only; note the quantity of OsO4 dissolved; quench the OsO4 solution by corn oil when the solution turned brown/black * Store unopened ampules only in locked filing cabinet; Nisansala has the key | | |
| **SECTION 7 – SPILL AND ACCIDENT PROCEDURES** | | |
| * Evacuate the laboratory immediately * Close the lab door(s) and post a “**NO ENTRY**” sign(s) explicitly mentioning the type of hazard * Cover the OsO4 spill with corn oil * 2% solution of OsO4 can be neutralized by twice its volume in vegetable oil * 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 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 OsO4 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; treat affected areas with corn oil * Have another person from the lab dial **911** if intense skin irritation is observed and specifically mention OsO4 exposure * Keep applying corn oil or 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 OsO4 exposure   *Inhalation*   * Move to fresh air * Dial **911** and inform emergency responders that the accident involved OsO4   **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 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 * 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](https://web2.uconn.edu/ehs/cwc/securereq/request.php) * P-Listed chemical. Empty containers must be managed as hazardous waste. * 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 copious amounts of water; use of surfactants is recommended. * Cover OsO4 spills with corn oil * 2% solution of OsO4 can be neutralized by twice its volume in vegetable oil | |
| Personal Hygiene | * Use standard chemical hygiene practices regarding PPE (see above). * Dispose of all disposable gloves after handling OsO4 * Wash hand thoroughly after handling OsO4 | |
| **SECTION 11 – SPECIFIC PROCEDURE** | | |
| A typical use of OsO4 in our laboratories is described in: Brückner, C.; Rettig, S. J.; Dolphin, D. ‘Formation of a *meso*-Tetraphenylsecochlorin and a Homoporphyrin with a Twist’, *J. Org. Chem.* **1998**, 63, 2094−2098.  CAUTION: Fume hood and eye protection required! It is best to scale your reaction so to be able to use an entire ampule of OsO4 to be opened and immediately dropped into the reaction flask.  *meso-Tetraphenyl-2,3-cis-dihydroxy-2,3-chlorin (****1****)*  *meso*-Tetraphenylporphyrin **4** (1.0 g, 1.63 × 10-3 mol) was dissolved/suspended in freshly distilled, ethanol-stabilized CHCl3/10% pyridine (200 mL) and was treated with OsO4 (540 mg, 1.3 equiv). The reaction flask was stoppered and stirred at ambient temperature for 4 days and was shielded from ambient light with aluminum foil. The reaction was then quenched by purging with gaseous H2S for 5 min (CAUTION: Fumehood, trapping of excess H2S!) (washing the organic reaction mixture with dilute base solutions to extract the H2S tends to form very stable emulsions). Following the addition of MeOH (20 mL), the precipitated black OsS was filtered off through Celite. The filtrate was evaporated to dryness by a stream of N2 or in vacuo. The resulting residue was loaded onto a silica gel column (25 × 7 cm) and eluted with CH2Cl2. The first fraction was starting material (400 mg, 40% recovery). MeOH/CH2Cl2 (1.5%) then eluted **1**. Slow evaporation from a MeOH/CH2Cl2 mixture gave **1** (520 mg, 8.02 × 10-4 mol, 49% yield) as a bright purple crystalline material. | | |

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| **SECTION 12A. APPROVAL** | | |
| I have reviewed, understood and agreed to follow this lab-specific standard operating procedure (LSOP) for osmium tetroxide*.* 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 for the use of osmium tetroxide. | | |
| **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.** | | |