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| **Brueckner Lab-Specific Standard Operating Procedure (LSOP):****Drying/Distilling of THF over Elemental Sodium and CH2Cl2 over NaH** |
| **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** |
| * THF – flammable solvent and forms highly flammable vapors with a wide explosion range
* THF and CH2Cl2 – cause skin irritation
* THF – may form an explosive peroxide
* Elemental Sodium (Na) and calcium hydride (CaH2) react violently with water, forming a caustic products – high risk of eye and skin damage
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| **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 – this includes the setup, refreshing, or quenching of the still
* Know the location of the nearest sand bucket (located in the between R413 and R415), eyewash, and safety shower before beginning work
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| **SECTION 3- ENGINEERING CONTROLS** |
| This set-up needs to be located in a secondary metal tray to limit a spill/fire if the spilled solvents catches fire.A water flow monitor needs to be attached that cuts the power to the heating mantle if not sufficient cooling is provided.Use of a blast shield in front of the set-up is required.Eliminate all sources (open flames, hot surface, static electricity and operation of mechanical and electrical equipment) near the setup during operation and particularly when dispensing solvents. |
| **SECTION 4 – WORK PRACTICES** |
| * Label each THF bottle with the date received, date opened and date last used.
* Test THF for peroxide before any distillation or purification of peroxide forming chemical.
* Minimize peroxide formation by storing in tightly sealed containers in a cool place in the absence of light.
* Never run the still without the proper engineering controls in place
* *Never attempt to dry a halogenated solvent with metallic sodium*.
* Become familiar with the cleaning/maintenance of the solvent still by asking more senior members in your research group and which part can be replaced/cleaned/decontaminated/degreased, and how.
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| **SECTION 5 – PERSONAL PROTECTIVE EQUIPMENT (PPE)** |
| * At a minimum, nitrile gloves, a lab coat, long pants as well as closed-toed footwear and chemical safety glasses must be worn when handling the solvent still.
* When performing a hazardous activity such as adding cutting sodium, or quenching sodium in the distillation pot, chemical splash goggles or a full-face shield in addition to the safety glasses must be use.
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| **SECTION 6 – STORAGE** |
| * The storage of flammable and combustible liquids in a laboratory, shop or building area must be kept to the minimum needed for research and/or operations.
* A can of sodium under mineral oil is located on the shelf next to the still, separated from other chemicals.
* Switch off the still entirely when not in use – including the cooling water.
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| **SECTION 7 – SPILL AND ACCIDENT PROCEDURES** |
| * In case of spills of large quantities of solvent (>500 mL), alert all occupants and evacuate the lab and consider your next move: Call 911, particularly when there is the risk of fire, or waiting until the solvent has evaporated, or a clean-up procedure, depending on the hazards the solvent poses.
* If safe to do so, turn off all sources of ignition.
* Turn off all sources of ignition – push the red emergency shut-off button near the exit.
* **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 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 solvent/sodium/sodium hydride/caustic base 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 while flushing.
* Have another person from the lab dial **911** if intense skin irritation is observed and specifically mention specific solvent/sodium/sodium hydride/caustic base exposure.

**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** |
| * Any solvents coming from the distillation pot without distillation must be quenched before being added to the waste containers.
* 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](http://ehs.uconn.edu/Regulated%20Waste%20Management/index.php)
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| **SECTION 10 – DECONTAMINATION PROCEDURES** |
| * Generally, all glassware can be cleaned with water/soap and acetone
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| **SECTION 11 – SPECIFIC PROCEDURE** |
| Solvents are distilled under a nitrogen atmosphere from a distillation flask containing metallic sodium/benzophenone (for non-halogenated solvents) or sodium hydride (for halogenated solvents). The refluxing solvent travels up the vacuum-insulated distillation column and condenses on the water-cooled condenser. The distillate collects at the top of the column in the distillation trap. The purified solvent is removed via syringe through the septum at the top or through the stopcock at the bottom of the distillation trap. Each of the stills is connected in series by water lines for the condensers and nitrogen lines for the inert atmosphere.**Setting-Up Solvent Stills*** The still is cooled down to room temperature. Ensure the heating mantle is turned off
* The solvent is poured into the round bottom flask which serves as the still pot to no more than 2/3 of its capacity.
* Add reducing and drying agents to the solvent.

**Solvent Still for Tetrahydrofuran*** In a fume hood, a small amount of metallic sodium is cut with a small knife into small pieces (gloves, dry surfaces, no aqueous solution open nearby), then the sodium is added to the solvent in the distillation flask.
* Add ~250 mg of benzophenone to the distillation flask, establish the inert atmosphere, and bring the solution to reflux for several hours to allow the sodium metal a chance to dry the solvent. When dry, the solution will have a deep blue to blue-green color (of the benzophenone ketyl species).
* If, after refluxing for several hours, the deep blue color does not develop, repeat previous two steps.
* Tetrahydrofuran form peroxides. Do not distill without adding a reducing agent such as sodium.

**Solvent distill for Dichloromethane*** A small amount of calcium hydride is added and the solution is refluxed under a N2 atmosphere for 4+ hours

**Maintenance and Inspections*** Check for the nitrogen flow at the bubbler and its level.
* Check water lines and conditions for weak spots and leaks.
* Check solvent level is at least 1/3 full.

**Cleaning of the Tetrahydrofuran or Dichloromethane Still*** After the solvent has cooled to room temperature, the distillation flask is moved to a fume hood.
* The residual solvent is carefully quenched with small amounts of 2-propanol added under a blanket of nitrogen over several hours and allowed to stand overnight, stir or swirl the mixture once in a while.
* The residual solvent is then carefully quenched with small amounts of absolute methanol over several hours until all the sodium metal is destroyed. Now carefully add excess water under nitrogen and properly dispose of the contents in the appropriate waste solvent canisters (non-halogenated/halogenated).

**Testing for Peroxides in THF*** In most safety literature, a conservative concentration of 100 ppm peroxides is used as a control point. All chemicals which are to be distilled must be tested prior to distillation regardless of age.
* Several methods are commonly used to detect for peroxide in the laboratory. Perhaps the most convenient method is use of peroxide test strips, manufactured by Aldrich and several other suppliers.
* For volatile organic chemicals, the test strip is immersed in the chemical for 1 second; then the tester breathes slowly on the strip for 15-30 seconds, or until the color stabilizes. The color is then compared to the scale provide on the packaging. Strip that offer a 1-100 ppm peroxide range are useful for determining if the material is below the control point of 100 ppm.
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| **SECTION 12A. APPROVAL** |
| I have reviewed, understand and agree to follow this lab-specific standard operating procedure (LSOP) for the operation of the solvent still for THF/Na and CH2Cl2/NaH *only*.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.)
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| **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.** |