#1 CONTACT INFORMATION:

<table>
<thead>
<tr>
<th>Procedure Title</th>
<th>Synthesis of Silatranes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Procedure Author</td>
<td>Skrypal, Vladislav / Suating, Paolo</td>
</tr>
<tr>
<td>Date of Creation/Revision</td>
<td>18 September 2015</td>
</tr>
<tr>
<td>Name of Responsible Person</td>
<td>Adler, Marc Jordan, PhD</td>
</tr>
<tr>
<td>Location of Procedure</td>
<td>LaTourette Hall 336</td>
</tr>
<tr>
<td>Approval Signature</td>
<td></td>
</tr>
</tbody>
</table>

#2 THIS STANDARD OPERATING PROCEDURE (SOP) IS FOR A:

- Specific laboratory procedure or experiment
- Generic laboratory procedure that covers several chemicals
- Generic use of specific chemical or class of chemicals with similar hazards

#3 PROCESS OR EXPERIMENT DESCRIPTION

This procedure is for the synthesis of 1-substituted silatranes from boratrane.

<table>
<thead>
<tr>
<th>Frequency:</th>
<th>☐ one time ☐ daily ☐ weekly ☐ monthly</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>☑ other: As needed</td>
</tr>
<tr>
<td>Duration per Expt:</td>
<td>ca. 8 hours</td>
</tr>
</tbody>
</table>

#4 SAFETY LITERATURE REVIEW & HAZARD SUMMARY

For assistance with this form contact NIU Environmental Health and Safety, 815-753-0404.
1. Boratranne – irritant; may cause irritation to skin and/or mucous membranes. Safety and toxicity of this material has not been fully investigated. Avoid prolonged exposure.

2. Aluminium (III) chloride (anhyd.) – corrosive; may be harmful if swallowed. Causes severe burns upon contact with skin and/or mucous membranes; suspected carcinogen and/or teratogen; severely nephrotoxic; very toxic to aquatic life.

3. Si-substituted triethoxysilanes – highly flammable liquid and vapour; corrosive – causes severe burns on contact with skin and/or mucous membranes; vapours are extremely harmful to eyes; suspected carcinogen and/or teratogen; fatal if ingested by mouth and/or inhalation.

4. Xylenes – highly flammable liquid and vapour; readily absorbed through skin and through respiration; confirmed carcinogen and teratogen; ototoxic; causes dizziness, drowsiness, and/or nausea; causes damage to internal organs; extremely toxic to aquatic life with lasting effects.

5. Si-substituted silatranes – may cause irritation to skin, eyes, and/or mucous membranes; suspected carcinogen and/or teratogen; convulsant; harmful if ingested by mouth.

#5 STORAGE REQUIREMENTS

1. Boratranne – to be used as soon as generated; store under partial vacuum in desiccator; keep away from moisture.

2. Aluminium (III) chloride (anhyd.) – store under partial vacuum in a desiccator set apart for anhydrous inorganics; keep away from moisture.

3. Si-substituted triethoxysilanes – store under partial vacuum in a desiccator set apart for reactive silanes. Keep under inert gas (N₂ or Ar). Keep away from moisture.

4. Xylenes – store in ‘Solvent’ cabinet; keep away from naked flames.

5. Si-substituted silatranes – store under partial vacuum in desiccator; keep away from moisture.

#6 STEP-BY-STEP OPERATING PROCEDURE

Steps to include in your procedure:

1. Don personal protective equipment.

☐ appropriate street clothing (long pants, close-toed shoes)

☐ nitrile gloves

☐ safety goggles ☐ safety glasses ☐ face shield

☐ lab coats

☐ other: ________________________

For assistance with this form contact NIU Environmental Health and Safety, 815-753-0404.
2. Check the location and accessibility of the safety equipment that serves your lab:

<table>
<thead>
<tr>
<th>ITEM</th>
<th>STATUS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laboratory Fume</td>
<td></td>
</tr>
<tr>
<td>Hood/Glove Box or other</td>
<td>Location: LaTourette 336</td>
</tr>
<tr>
<td>Ventilation Control</td>
<td></td>
</tr>
<tr>
<td>Eyewash/Safety Shower</td>
<td>Location: LaTourette 336</td>
</tr>
</tbody>
</table>

3. To a 500 mL flame/oven round-bottom flask with stir bar and fitted with a Dimroth condenser and flushed with argon add boratrane and 200 mL xylenes, Si-substituted triethoxysilane, and a catalytic amount of aluminium (III) chloride.

4. Heat round-bottom flask to 135 °C with stirring for 3 to 4 hours, keeping contents under argon.

5. Stop heating and let cool. Filter solids under vacuum; keep solids, discard filtrate appropriately.

6. Recrystallize solids via hot recrystallization with xylenes. Discard solids and let silatrane crystals form in filtrate overnight, plunging flask in ice bath if necessary.

7. Dispose of hazardous solvents, solutions, mixtures, and reaction residues as hazardous waste. See EH&S Hazardous Waste Program
   http://www.ehs.niu.edu/ehs/chemical/waste.shtml

6. Clean up work area and lab equipment.

7. Remove PPE and wash hands.

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**#7 WASTE DISPOSAL**

Typical waste disposal; used xylenes (ca. 500 mL) and excess boratrane to be disposed in appropriate waste container for non-halogenated solvents.

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**#8 TRAINING REQUIREMENTS**

General Training *(check all that apply):*

- [✓] General Safety & Emergency Preparedness
- [✓] Chemical Safety for Laboratories
- [ ] Radiation Safety
- [ ] Biosafety training
- [ ] Other: ____________________________

Location Where Records Maintained: www.niu.edu/chembio/labsafety/pdfs

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For assistance with this form contact NIU Environmental Health and Safety, 815-753-0404.
Laboratory-specific training *(check all that apply):*
- [ ] Review of SDS for other chemicals involved in process/experiment
- [ ] Review of this SOP
- [ ] Other: ___________________________

| Location Where Records Maintained: | www.niu.edu/chembio/labsafety/pdfs |

| #9 PRIOR APPROVALS |

Prior approvals are required by the following University Committees:

- **Radiation Safety Committee:** Radioactive material
- **Radiation Safety Committee:** X-Ray machines
- **Laser safety:** Laser producing equipment Class 3b or above.
- **IACUC:** Animal use in research
  - [http://www.orc.niu.edu/orc/animal_research/index.shtml](http://www.orc.niu.edu/orc/animal_research/index.shtml)
- **IBC:** Recombinant DNA, potential pathogens, human tissue/body fluids
  - [http://www.orc.niu.edu/orc/biosafety/niupolicy.shtml](http://www.orc.niu.edu/orc/biosafety/niupolicy.shtml)