# CONTACT INFORMATION:

<table>
<thead>
<tr>
<th>Procedure Title</th>
<th>Synthesis of Boron nitride based nanomaterials</th>
</tr>
</thead>
<tbody>
<tr>
<td>Procedure Author</td>
<td>Vijayaraghavan Kalavakunda</td>
</tr>
<tr>
<td>Date of Creation/Revision</td>
<td>08/26/2015</td>
</tr>
<tr>
<td>Name of Responsible Person</td>
<td>Prof. Hosmane</td>
</tr>
<tr>
<td>Location of Procedure</td>
<td>FR 406</td>
</tr>
<tr>
<td>Approval Signature</td>
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# THIS STANDARD OPERATING PROCEDURE (SOP) IS FOR A:

- Specific laboratory procedure or experiment
  Examples: synthesis of Boron nitride nanotubes and derivatives.
- Generic laboratory procedure that covers several chemicals
- Generic use of specific chemical or class of chemicals with similar hazards

# PROCESS OR EXPERIMENT DESCRIPTION


<table>
<thead>
<tr>
<th>Frequency:</th>
<th>one time</th>
<th>daily</th>
<th>weekly</th>
<th>monthly</th>
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<tr>
<th>Duration per Expt:</th>
<th>_________ minutes; or 12 hours</th>
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</table>
1. **Ammonium tetrafluoborate** - Corrosive solid. Can cause severe eye damage and skin burn. Personal protective equipment (PPE) - Gloves, safety glasses, and lab coat.

2. **Lithium borohydride** - Toxic chemical, emit flammable gases when in contact with water or moisture, corrosive and eye irritant. (PPE) - Gloves, safety glasses, lab coat.

3. **Sodium trinitride** - Highly toxic by injection and skin absorption. (PPE) - Gloves, safety glasses, lab coat.

4. **Lithium** - When in contact with water emits flammable gases, skin corrosion, and serious eye damage. (PPE) - Gloves, safety glasses, lab coat.

5. **Zinc powder** - No immediate health hazard. Avoid disposal to the environment. Please use appropriate waste disposal. (PPE) - Gloves, safety glasses, lab coat.

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**STORAGE REQUIREMENTS**

1. **Ammonium tetrafluoborate** - Store in cool place. Keep container tightly closed in a dry and well-ventilated place. Moisture sensitive. Keep inside a glove box.

2. **Lithium borohydride** - Store in cool place. Keep container tightly closed in a dry and well-ventilated place. Moisture sensitive, do not allow water to enter container. Keep inside a glove box.

3. **Sodium trinitride** - Keep container tightly closed in a dry and well-ventilated place. Keep inside the glove box.

4. **Lithium** - Keep container tightly closed in a dry and well-ventilated place. Keep inside the glove box.

5. **Zinc powder** - Keep container tightly closed in a dry and well-ventilated place. Keep inside the glove box.

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**STEP-BY-STEP OPERATING PROCEDURE**

**Reaction Setup:**

1. All the equipment required to perform this experiment are gathered in the glove box. The list includes:
   i). Beaker (250ml)
ii). 5 spatulas  
iii). Glass funnel  
iv). Mortar and Pestle  
v). Pressure vessels, lid, screws and washer  
vii). Any chemical if not already present inside the glove box.

2. The SOP for glove box is used to take all the listed items inside the glove box.

3. The chemicals are weighed in the beaker, the scale set to tare each time a new material is weighed.
   a. Ammonium tetrafluoroaborate - 10.484g  
   b. Lithium borohydride - 2.178g  
   c. Sodium trinitride - 6.501g  
   d. Lithium - 0.7g  
   e. Zinc powder - 6.53g

4. After all the chemicals are weighed in the beaker they are transferred to the mortar and ground well using the pestle.

5. After through grinding into a fine powder the contents are transferred into the pressure vessel using a funnel.

6. The pressure vessel is removed from the glove box following glove box SOP and then it is set up for heating inside the furnace.

7. The furnace manufacturer’s SOP is used for setting up the furnace.

8. The glassware and other equipment are cleaned.

9. The PPE is removed and hands are washed.

Chemical reaction:

$$2 \text{LiBH}_4(s) + 2\text{NH}_4\text{BF}_4(s) + 8\text{NaN}_3(s) \rightarrow (\text{B-N})_2(s) + 2\text{Li}(s) + 11\text{N}_2(g) + 8\text{H}_2(g) + 8\text{NaF}(s)$$

When a finely powdered mixture of LiBH$_4$ (100mmol), NH$_4$BF$_4$ (100mmol) and NaN$_3$ (400mmol) is pyrolyzed the generated gas pressure within 100 mL capacity pressure vessel is calculated (P = nRT/V) to be 836.9 atm that is far below the pressure limit of 6000 atm as certified by the manufacturer of the pressure vessel, Meyer Tool & Manufacturing Company, Oak Lawn, Illinois 60453.

Safety Limit:

1. It is essential that the generated pressure inside the Pressure Vessel must be far less than the maximum specification certified by the manufacturer!

2. Do not vary from amounts in this SOP.
Purification of product:
1. On completion of the reaction the pressure vessels are removed from the
furnace and opened using appropriate tools inside a fume hood.
2. The contents are transferred into a clean dry beaker and the amount of crude
mixture is weighed.
3. Measured amounts of deionized water is slowly added to the mixture and
stirred for an hour. The contents are filtered using a vacuum filtration setup.
4. The contents are washed thrice. The contents are then transferred into a
beaker and 3M HCl is added slowly drop by drop over a period of 2 hours. The
procedure is repeated till the decant turns colorless.
5. The products are centrifuged and washed with deionized water till the solution
turns neutral.
6. The product is further centrifuged using acetone and the contents are collected
in a dry pre weighed round bottom flask.
7. The acetone is evaporated in a “rotavap” and the product is dried under high
vacuum overnight at room temperature.

Additional Procedures:
1. The specific pressure vessels provided by “Amseng” alone needs to be
used.
2. The furnace log sheet must be completed with start and end timings
recorded and the door should be locked with the pad locks provided till
the furnace is on.
3. Under emergency the procedure needs to be SHUT OFF using ELECTRIC
POWER BRAKER #23 provided outside the room.

Steps to include in your procedure:

1. Don personal protective equipment.

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

☒ gloves; indicate type: Nitrile

☒ safety goggles ☒ safety glasses ☒ face shield

☒ lab coats

☐ other: ________________________________

2. Check the location and accessibility of the safety equipment that serves your lab:

<table>
<thead>
<tr>
<th>ITEM</th>
<th>STATUS</th>
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<tbody>
<tr>
<td>Laboratory Fume</td>
<td></td>
</tr>
<tr>
<td>Hood/Glove Box or other Ventilation Control</td>
<td>Location: FR 301 and FR 304</td>
</tr>
<tr>
<td>Eyewash/Safety Shower</td>
<td>Location: FR 301 and FR 304</td>
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#7 WASTE DISPOSAL

All experimental waste goes to the inorganic waste container.

#8 TRAINING REQUIREMENTS

**General Training (check all that apply):**

- General Safety & Emergency Preparedness
- Chemical Safety for Laboratories
- Radiation Safety
- Biosafety training
- Other: ___________________________

Depending on the hazardous materials and processes you will be working with in this SOP, additional safety training may be required by NIU. [PRECEDING GUIDANCE TEXT MAY BE DELETED]

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<th>Location Where Records Maintained:</th>
<th>FR 301 Front Desk</th>
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**Laboratory-specific training (check all that apply):**

- Review of SDS for other chemicals involved in process/experiment
- Review of this SOP
- Other: ___________________________

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#9 PRIOR APPROVALS

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For assistance with this form contact NIU Environmental Health and Safety, 815-753-0404.