Safe Work Procedure

**Process/Equipment:** CEM Discover Microwave Synthesiser

**Location:** 18.214

**Procedure Developed by:** Marc Bouillon, Sandra Chapman

**Approved by:** Prof. Stephen Pyne

**Date:** 12th Feb 2008

**Referenced UOW Guidelines, legislation, codes of practice, Australian Standards etc:**
- PER-OHS-GUI-048.4 Laboratory Safety Guidelines
- CEM Discover Operation Manual

**Personal Protective Equipment Required (Check the box for required PPE):**
- Appropriate gloves
- Safety glasses
- Covered shoes
- Labcoat

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**! Pure metals (e.g. copper, iron, zinc etc) should not be used in the microwave synthesiser as these can lead to arcing or to localized heating and explosion of the reaction vessel.**

**! Use of metal salts (Pd(OAc)2 etc) should be kept at the 5 mol% level.**

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<thead>
<tr>
<th>Step</th>
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<th>Hazards Identified</th>
<th>Risk Score</th>
<th>Controls</th>
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</table>
| 1    | Preparation of the reactants and reaction flask  
  a) Complete a risk assessment on all chemicals and solvents used and have it signed by your supervisor.  
  b) Prepare reaction vessel with reactants in your research laboratory  
  c) Stopper the reaction vessel  
  d) Transport the reaction vessel to 18.214 | Reaction may proceed unexpectedly, resulting in uncontrolled reaction and equipment damage  
  Spillage or exposure to reactants during preparation or transport  
  Breakage of reaction vessel during handling or transport | Medium (9)  
  Medium (12)  
  Medium (12) | Complete a small scale test reaction (1ml), standard reaction volume in sealed vessel is 3mL  
  Prepare reaction vessel in the fume hood  
  Ensure reaction vessel is stoppered to transport to 18.214  
  Handle glass reaction vessel with care. Do not use if cracked or damaged. Immediately clean up any spills |
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| 2    | Setup microwave synthesiser software for the reaction  
   a) Switch on the microwave and then switch on the computer  
   b) Select microwave user by double clicking on Synergy  
   c) Select your username (advisable to go to Mode to turn off explorer)  
   d) First Time Users (with the system administrator), set up your general preferences These are not the values for your reaction but are defaults that will be called up each time you use this method.  
   e) Set up methods appropriate for your reaction and save the method under your username  | Fume-hood not working correctly resulting in exposure to volatile material  
   Experimental limits which set in preferences are not set to stop high, undesirable pressure or temperature if method is incorrect for reactants  
   Incorrect parameter settings as there is currently no cooling unit attached to the synthesiser  
   Incorrect method settings  
   Open vessel reaction is selected under method parameters, when closed vessel reaction will be commenced This would result in continued heating, without pressure, monitoring and possible pressurisation of reaction vessel | Medium(9)  
   Medium (13)  
   Medium (12)  
   Medium (12)  
   Medium (9) | Contact B&G service centre on extension 3217 to report fume-hood malfunction. Do not carry out any open vessel reactions until repaired.  
   New users should set the following recommended preferences:  
   Prestirring 30s  
   Pressure 5 bar  
   Power 20 W  
   Stirring high  
   Reactions greater than 1 -2 hours are not recommended for new users  
   Ensure that power Max is set to OFF  
   Consult MSDS for reactant and with Supervisor as to the appropriate temperature, pressure, power and reaction time. Ensure risk assessment completed.  
   Ensure correct parameters are entered into method.  
   Always check that open vessel is deactivated when carrying out a closed vessel reaction |
| 3a   | Set up microwave synthesiser for: closed vessel reaction  
   a) Place reaction vessel into microwave  
   b) Put IntelliVenr Attenuator and IntelliVent pressure sensor on top of vessel  
   c) Lock sample compartment los  | Exposure to volatile components in reaction mixture  
   Reaction vessel breaks in microwave, spillage of reaction mixture | Medium (12)  
   Medium(12) | Ensure the fume-hood is ON  
   Ensure that cap is on for closed vessel reaction  
   Check the integrity of reaction vessel before reaction commenced  
   Ensure spillage beaker is in place when setting up instrument |
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<td><strong>open vessel reaction</strong>&lt;br&gt;a) Ensure method parameters are selected for open vessel reaction&lt;br&gt;b) Place spill cup and spacer into synthesiser (ensure aligned correctly)&lt;br&gt;c) Place 100ml RB flask containing reaction mixture onto spacer&lt;br&gt;d) Place open vessel attenuator into synthesiser and click into place&lt;br&gt;e) Position condenser rod behind reaction compartment&lt;br&gt;f) Place glass adapter onto RB flask&lt;br&gt;g) Clamp glass condenser and place onto glass adapter&lt;br&gt;h) Connect condenser tubing to water supply and turn on water</td>
<td>Temperature sensor does not sit into spillage beaker or spacer correctly&lt;br&gt;Reaction will not commence&lt;br&gt;Error message that there is an open door&lt;br&gt;Exposure to fumes as condenser is not connected</td>
<td>Low (14)&lt;br&gt;Insignificant (16)&lt;br&gt;Medium (9)</td>
<td>Ensure spacer gap is positioned directly over hole in spillage beaker for the temperature sensor&lt;br&gt;Ensure open vessel reaction has been selected.&lt;br&gt;Check that water is going through condenser before reaction commenced.&lt;br&gt;Use a vent vessel if necessary – as shown below</td>
<td></td>
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</table>
| **Commence reaction**<br>a) Press play<br>b) Monitor reaction progress, temperature and pressure on screen (note the nature of the reaction and solvents will determine how quickly set temperature and pressure is reached) | Reaction does not proceed<br>Equipment switches off during run and reaction vessel is removed resulting in injury<br>Reaction is not proceeding as expected, resulting in dangerously high temperature and/or pressure in the closed reaction vessel which results in damage to equipment | Insignificant (16)<br>Medium (12)<br>Medium (9) | The reaction vessel and contents may still be at a high temperature and/or pressurised. Allow reaction vessel to cool down before removing from synthesiser.<br>Immediately stop the reaction by
1. Selecting the stop button on the software (this will shut everything down)<br>2. Selecting pause on software (this will allow pressure and temperature to be reset so experiment can be recommenced)<br>3. Pressing the stop button on the microwave synthesiser.<br>Do not move reaction vessel until it has cooled down. Monitor the temperature on the screen, the pressure |
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<td></td>
<td></td>
<td>Temperature and pressure are above release limits but the reaction vessel is removed</td>
<td>Medium (12)</td>
<td>will no longer be recorded if reaction has been stopped. To release the closed reaction vessel, a window will appear that asks <em>Parameters above release limits</em>, release Y/N. Do not select Y unless you are sure of the boiling point of the solvents and risk free conditions if the vessel is removed.</td>
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<tr>
<td>5.</td>
<td>a) Reaction completed, commence cooling</td>
<td>Handling of hot, pressurised reaction vessel, resulting in burns, spillage or breakage</td>
<td>Medium (9)</td>
<td>Reaction vessels should be cooled for a minimum of 20 minutes before they removed from synthesiser. Pressure should be below 1 bar before release. The microwave synthesiser will automatically release vessel based on preferences set. Hold reaction vessel at top when removing from synthesiser or transporting</td>
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<tr>
<td></td>
<td>b) Remove IntelliVent, reaction vessel and attenuator from synthesiser</td>
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<td></td>
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<td></td>
<td>c) Close program and shut down computer</td>
<td></td>
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<tr>
<td></td>
<td>d) Switch off synthesiser</td>
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<td></td>
<td>e) Carry reaction vessel to research laboratory for analysis</td>
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Risk Ranking

The outcome of a risk ranking exercise is the subjective ranking of significance for each identified risk. Action needs to be considered in direct relation to the ranked significance of risk. As well as an individual risk score, each risk is also placed within one of three broad categories –

- **Extreme Risk** – The procedure should not take place until the risk has been lowered
- **High Risk** – an unacceptable level of risk, which calls for control measures to be implemented immediately
- **Moderate Risk** – a risk requires action to reduce the risk, within reasonable timescales
- **Low Risk** – Perhaps acceptable risk, where current controls appear adequate, should be monitored to ensure no lowering of control effectiveness occurs.
- **Negligible Risk** – Acceptable risk in most instances which should be reviewed regularly to ensure controls are adequate

Risk Ranking matrix allows a risk ranking number to be assigned to a specific risk.

<table>
<thead>
<tr>
<th>Consequence</th>
<th>Major</th>
<th>Moderate</th>
<th>Minor</th>
<th>Insignificant</th>
</tr>
</thead>
<tbody>
<tr>
<td>Common, Expected</td>
<td>1</td>
<td>2</td>
<td>4</td>
<td>7</td>
</tr>
<tr>
<td>Likely, could occur</td>
<td>3</td>
<td>5</td>
<td>8</td>
<td>11</td>
</tr>
<tr>
<td>Possible but not often</td>
<td>6</td>
<td>9</td>
<td>12</td>
<td>14</td>
</tr>
<tr>
<td>Unlikely, rare</td>
<td>10</td>
<td>13</td>
<td>15</td>
<td>16</td>
</tr>
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</table>

Table 1: Risk Ranking Matrix

**Likelihood**

Common – is expected to occur in most circumstances
Likely – will probably occur in most circumstances
Possible – Might occur at some time
Unlikely, Rare – May occur only in exceptional circumstances

**Consequences**

Major – Extensive, Serious Injury, Fatality > $250K
Moderate – Medical Treatment required, Lost time injury, $50K - $250K
Minor – Minor Injury, First Aid Treatment, $5K - $50K
Insignificant – No injury, < $5K