Model Assessment

Hazardous Substances Policy - Assessment

CHEMICAL HAZARD AND RISK ASSESSMENT

<table>
<thead>
<tr>
<th>School/Dept</th>
<th>Chemistry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Assessor</td>
<td>AN Other</td>
</tr>
<tr>
<td>Assessment Number</td>
<td>Ch1AFD</td>
</tr>
<tr>
<td>Date of Assessment</td>
<td>10/08</td>
</tr>
</tbody>
</table>

Notes
Guidance on making an assessment is given in Chemical Hazard and Risk Assessment (GUIDANCE/22/CHRA/05).
Guidance is also available from the attached Guidance on Completing the Chemical Hazard and Risk Assessment Form.
Substance data is available in HAZDAT. Use a continuation sheet or word processor to expand any section of this form.
An MS Word file for this form is available from http://www.hsu.bham.ac.uk/univ/hspolicy/hs15/HS2ASSFM.DOC.

1. LOCATION OF THE WORK ACTIVITY
Chemistry West

2. PERSONS WHO MAY BE AT RISK
List names where possible
AN Other

3. ACTIVITY ASSESSED
Synthesis of Acyl Ferrocene Derivative

4. MATERIALS INVOLVED
Attach copies of data sheet(s)

<table>
<thead>
<tr>
<th>NAME and CAS NUMBER</th>
<th>AMOUNT and FORM</th>
<th>HAZARD</th>
<th>RISK PHRASES</th>
<th>REPORTABLE?</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ferrocene 102-54-5</td>
<td>6g, solid</td>
<td>Highly Flammable. Harmful.</td>
<td>Highly flammable. Harmful if swallowed.</td>
<td>N</td>
</tr>
<tr>
<td>Dichloromethane 75-09-2</td>
<td>200 ml, liquid</td>
<td>Harmful</td>
<td>Limited evidence of a carcinogenic effect (Category 3 carcinogen) Can be absorbed through the skin</td>
<td>N</td>
</tr>
<tr>
<td>Aluminium chloride 7446-70-0</td>
<td>6g, solid</td>
<td>Corrosive</td>
<td>Causes burns.</td>
<td>N</td>
</tr>
<tr>
<td>Magnesium sulphate 7487-88-9</td>
<td></td>
<td></td>
<td>Not hazardous, except as inert dust</td>
<td></td>
</tr>
<tr>
<td>Cyclohexane 110-82-7</td>
<td>10 ml, liquid</td>
<td>Highly Flammable. Harmful. Dangerous for the environment</td>
<td>Highly flammable. Irritating to skin. Harmful: may cause lung damage if swallowed. Vapors may cause drowsiness and dizziness. Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.</td>
<td>N</td>
</tr>
</tbody>
</table>

If substance is reportable, have you reported it to the Health and Safety Unit? YES/NO (see Note 4)
**INTENDED USE and JUSTIFICATION (where appropriate)**

Give brief details and attach protocol/instructions. Justification is needed for exceptionally hazardous substances (see Note 5).

1. **Dispensing and preparation of reagents:** ferrocene (3g) in dichloromethane (15ml); aluminium chloride (5.5 g), acetyl chloride (3.5ml) in dichloromethane (20ml); dichloromethane (5 x 10ml), (3 x 10ml), magnesium sulphate, cyclohexane (10ml)

2. **Synthesis:** Under an atmosphere of dry nitrogen, ferrocene in dry dichloromethane is added dropwise over a period of 15 minutes to a stirred mixture of aluminium chloride and acetyl chloride in dry dichloromethane. The resulting mixture is stirred at room temperature for two hours then cooled in an ice bath to 0 °C and ice is carefully added to decompose the excess starting materials.

3. **Work up:** The mixture is filtered and the solid residue washed with dichloromethane until colourless. The red solution is transferred to a separating funnel and separated from the aqueous phase. The aqueous phase is subsequently washed several times with dichloromethane and the combined red organic phases dried over anhydrous magnesium sulphate. After filtration the red solution is reduced in volume to approximately 20ml using a rotary evaporator and at this point cyclohexane (10ml) is added. The solution is again concentrated to a volume of ~20ml and allowed to cool. Filter off the crystals using a Büchner funnel and air dry the solid by sucking air through the funnel for 5-10 minutes.

**RISKS to HEALTH and SAFETY from INTENDED USE**

From personal exposure or hazardous reactions. Refer to WELs, flash points, etc., as appropriate. Are pregnant women, breast-feeding mothers especially at risk?

1. **Dispensing and preparation of reagents:**
   - **Ferrocene** is a very low volatility, non-dusty solid. Dispensing 3g at room temperature not expected to cause significant exposure. It is highly flammable, no fire risk during dispensing as it will not be in contact with an ignition source.
   - **Dichloromethane** is a very volatile liquid, vp 336mmHg, a category 3 carcinogen and can be absorbed directly through the skin. Risk of inhalation when dispensing from winchester. Skin contact not likely, but risk from splashing if spilt.
   - **Aluminium chloride** is a non-volatile solid, corrosive to tissue and reacts with water. Small risk of skin exposure if spilt during weighing/transfer. Insignificant risk of exposure via inhalation or eye contact as not airborne. Insignificant reaction risk as no water present.
   - **Acetyl chloride** is a very volatile liquid, vp 353mmHg, highly flammable - fp 5°C, corrosive to tissue and reacts violently with water. Risk of inhalation when dispensing from winchester. Significant skin contact not likely, but risk from splashing if spilt. Risk of fire from vapour in event of spill when decanting. Insignificant reaction risk as no water present.
   - **Magnesium sulphate** is a non hazardous solid and significant exposure to dust not expected during dispensing.

2. **Synthesis:** Initial mixture carried out at room temperature in a closed flask – small quantities, max 35ml dichloromethane, 3.5ml acetyl chloride – very low inhalation and skin contact risks. Un-reacted starting materials deactivated under controlled conditions, with ice at 0°C – temperature below flash point so low risk of fire if vapour comes into contact with ignition source. Low temperature also reduces volatility so inhalation risk reduced. Ice added slowly to cooled mixture reduces risk of reaction to water.

3. **Work up:** Dichloromethane used in 10ml lots - excessive exposure to vapour unlikely so inhalation risk low. 10ml Cyclohexane small quantity means exposure to vapour unlikely so inhalation risk low, but may ignite on contact with a very hot surface.

**CONCLUSIONS ABOUT RISKS**

Is level of risk acceptable? Can risk be prevented or reduced by change of substance/procedure? Are control measures necessary?

Unacceptable inhalational risk and possible risk of skin contact to dichloromethane, acetyl chloride and cyclohexane during stage 1.

Reaction risk if acetyl chloride and aluminium chloride come into contact with water.

Fire risk if cyclohexane comes into contact with an ignition source.
Model Assessment

**8 CONTROL MEASURES**

Additional to Good Chemical Practice, e.g., fume cupboard, etc. Any special requirements, e.g., glove type, etc.

1. **Dispensing and preparation of reagents:**
   - Fume cupboard required for dispensing dichloromethane, acetyl chloride and cyclohexane.
   - Lab coat and safety glasses to be worn. Nitrile gloves required for skin protection against splashes.
   - Keep acetyl chloride and aluminium chloride out of contact with water.
   - Remove hot surfaces from area when handling cyclohexane.

2. **Synthesis:**
   - Lab coat and safety glasses to be worn. Nitrile gloves required for skin protection against splashes.
   - Ensure mixture is cooled to 0°C and add ice slowly.
   - Keep acetyl chloride and aluminium chloride out of contact with water.

3. **Work up:**
   - Lab coat and safety glasses to be worn. Nitrile gloves required for skin protection against splashes.
   - Keep acetyl chloride and aluminium chloride out of contact with water.
   - Remove hot surfaces from area when handling cyclohexane.

**9 INSTRUCTION/TRAINING**

Specify course(s) and/or special arrangements.
- Safe use of rotary evaporator
- Waste disposal
- Correct use of fume cupboard
- Laboratory chemical safety course

**10 MONITORING**

Performance of control measures.
- Face velocity of fume cupboard. Condition of gloves.
- Personal exposure
- Health Surveillance, specify measures agreed with Health and Safety Unit
  - N/A

**11 WASTE DISPOSAL PROCEDURE**

Include name, 6-digit code and H numbers if to be sent away for disposal.
- Red organic solution from reaction mixture
  - 14 06 02 other halogenated solvents and solvent mixtures
  - H3A, H6
- Aqueous waste can be washed down sink.

**12 REVIEW**

Enter the date or circumstances for review of assessment (maximum review interval 5 years)
- 1 year

**13 EMERGENCY ACTION**

**TO CONTROL HAZARDS**
- To stabilize situation e.g., spread absorbent on liquid spill, etc.
  - Liquids: Use inert spill absorbent to limit spread of spillage, eliminate sources of ignition
  - Solids: avoid contact with water.

**TO PROTECT PERSONNEL**
- Evacuation, protection for personnel involved in clean-up. Special First Aid
  - Liquid spills: Keep personnel not involved in clean up out of area. Evacuate area if large spill
  - PPE for clean-up: Lab coat, safety glasses, viton gloves. Large spills; type B RPE filter for acetyl chloride, type AX RPE filter for dichloromethane, type A RPE filter for cyclohexane

**TO RENDER SITE OF EMERGENCY SAFE**
- Clean-up/decontamination
  - Scoop up contaminated spill absorbent and place in a suitable, labelled container.
  - Scoop up solid spill and place in a suitable, labelled container.
  - Thoroughly wash affected area with water.
  - Thoroughly ventilate site of spillage

**14 EMERGENCY CONTACT**

NAME: AN Other
PHONE: 4XXXX