**HAZARD IDENTIFICATION, RISK ASSESSMENT & CONTROL**

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| NAME: Klementine Burrell-Sander  | DATE: 19.04.21 | **OVERALL RISK RATING** |
| WORKBOOK AND PAGE REFERENCE:  |  | **Medium** |

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| **ACTIVITY SUMMARY** |
| REACTION SCHEME AND EQUATION / DESCRIPTION OF PROCESS**Reduction of ferrocenyl methyl ester (to give ferrocenylmethanol)**1. Add powdered LiAlH4 (0.20 g, 1.75 equiv.) to ice-cold dry THF (2.5 mL) under nitrogen in a 100 mL 3-necked flask with a reflux condenser
2. Add a solution of distilled ferrocenylmethyl ester (1.00 g, 3.03 mmol) in THF (0.8 mL) dropwise
3. Allow mixture to come to room temperature and heated to 45-50 C for 1 hour
4. Upon completion of the reaction, pour in small portions into ice-cooled EtOAc (2.5 mL) with vigorous stirring. Temporary thickening of the mixture should occur upon each addition
5. Carefully add water in small portions (1.8 – 2 mL)
6. The inorganics should separate as a compact, creamy material and the brown organic phase can be removed by decantation
7. Rinse inorganics thoroughly with EtOAc (5 x 0.2 mL)
8. Dry combined organic phases with K2CO3 (3 x 0.25 g, pearls) with stirring
9. Concentrate brown organic phase under reduced pressure until total volume is ~ 2 mL
10. Crystallise (scratch with spatula and cool in ice bath)
11. Filter off yellow powdery solid
12. Wash solid with toluene and dry *in vacuo*
13. Remove volatiles from mother liquor and recrystallise remaining solid from toluene (1g/7mL solvent)
14. Product should be a yellow solid (yield = 85%)
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| **CHEMICAL HAZARDS** |
| **REAGENTS /INTERMEDIATES/ PRODUCTS/ WASTE** | **Quantity (mL or g)** | **Size of reagent bottle (L or kg)** | **Quantity (mmoles or equivalents)** | **GHS HAZARD CLASSES AND CATEGORIES (refer to SDS)** | **L\*** | **C\*** | **RISK RATING** |
| Ferrocenyl methyl ester  | 1.00 g |  | 3.03 mmol | H228 Flammable solid.H302 + H332 Harmful if swallowed or if inhaled.H361 Suspected of damaging fertility or the unborn child.H373 May cause damage to organs (Liver) through prolonged orrepeated exposure if inhaled.H410 Very toxic to aquatic life with long lasting effects**DG 4.1 - flammable solid** | **U** | **Mi** | **L** |
| LiAlH4 | 0.2 g | 25 mL  | 5.27 mmol | H225 Highly flammable liquid and vapor.H302 Harmful if swallowed.H315 Causes skin irritation.H318 Causes serious eye damage.H335 May cause respiratory irritation.H336 May cause drowsiness or dizziness.H351 Suspected of causing cancer.**DG 4.3 – gives off flammable gas in contact with water**  | **U** | **Mo** | **M** |
| THF | 3.5 mL  | 2.5 L  |  | H225 Highly flammable liquid and vapor.H302 Harmful if swallowed.H319 Causes serious eye irritation.H335 May cause respiratory irritation.H336 May cause drowsiness or dizziness.H351 Suspected of causing cancer.**DG 3 – flammable liquid**  | **U** | **Mi** | **L** |
| Ethyl acetate  | 3.5 mL  | 2.5 L |  | H225 Highly flammable liquid and vapor.H319 Causes serious eye irritation.H336 May cause drowsiness or dizziness**DG 3 – flammable liquid** | **U** | **Mi** | **L** |
| Toluene  | 10 mL  | 2.5 L  |  | H225 Highly flammable liquid and vapor.H304 May be fatal if swallowed and enters airways.H315 Causes skin irritation.H336 May cause drowsiness or dizziness.H361 Suspected of damaging fertility or the unborn child.H373 May cause damage to organs (Central nervous system) throughprolonged or repeated exposure.**DG 3 – flammable liquid** | **U** | **Mi** | **L** |
| K2CO3  | 0.75 g  | 500 g  |  | H315 Causes skin irritation.H319 Causes serious eye irritation.H335 May cause respiratory irritation. | **U** | **Mi** | **L** |
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**Restricted, prohibited and high-risk chemicals (as described in** [**Section 5.8**](https://au-mynotebook.labarchives.com/share/SAFETY%2520NOTES/NDQuMnw1NjEwMS8zNC9UcmVlTm9kZS8xODcwNDQyMDIyfDExMi4xOTk5OTk5OTk5OTk5OQ%3D%3D) **of the School Safety Handbook) require additional safety documentation prior to use. If this is applicable to any of the chemicals above, ensure that the University’s Risk Assessment for High-Risk Chemicals is also completed.**

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| **HAZARD ASSOCIATED WITH THE ACTIVITIES (\*L = LIKELIHOOD; C = CONSEQUENCE. REFER TO RISK MATRIX ON PAGE 3)** |
| **PROCESSES/TECHNIQUES** | **Is SWP** [**available**](https://au-mynotebook.labarchives.com/share/SAFETY%2520NOTES/OS4xfDU2MTAxLzcvVHJlZU5vZGUvNDI0NzQ5NjM3NHwyMy4wOTk5OTk5OTk5OTk5OTg%3D)**?** | [**SWP training**](https://canvas.sydney.edu.au/courses/19009/pages/safe-work-procedures?module_item_id=640117) **completed?** | **POTENTIAL HARM (What could go wrong?)** | **L\*** | **C\*** | **RISK RATING** |
|  | **Yes** | **NO** | **Yes** | **No** | **N/A** |  |  |  |  |
| Stirring/mixing |[x] [ ] [x] [ ] [ ]  Chemical splash and subsequent chemical exposure to skin/eyes, inhalation of chemicals  | **U** | **Mi** | **L** |
| Rotary evaporation/distillation |[x] [ ] [x] [ ] [ ]  Spillage of chemicals and subsequent exposure to skin/eyes, inhalation, broken glassware, electric shock from machine | **U** | **Mi** | **L** |
| Heating |[x] [ ] [x] [ ] [ ]  Burns from hotplate/hot glassware, spillage of hot chemicals, chemical exposure, shattered glassware  | **U** | **Mi** | **L** |
| Choose an item. |[ ] [ ] [ ] [ ] [ ]   | **-** | **-** | **-** |
| Choose an item. |[ ] [ ] [ ] [ ] [ ]   | **-** | **-** | **-** |

**\*Likelihood: AC = Almost Certain, L = Likely, P = Possible, U = Unlikely, R = Rare; Consequence: In = Insignificant, Mi = Minor, Mo = Moderate, Ma = Major, S = Severe.**

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| **EQUIPMENT AND CONDITIONS** |
| LIST ANY EQUIPMENT REQUIRED: | Is there an SWP associated with the equipment being used? **Yes ☐** SWP training for use of equipment completed? **Yes ☐** Has the equipment been modified? **No ☐**; if yes, who has inspected and approved the modification? Name: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Total volume of reagents and solvents\*:Vessel capacity:Max/Min Temp: \_\_\_**0 – 50**\_\_\_\_\_\_\_\_ °C Max/Min Pressure: \_\_\_\_\_\_**0.1 – 1** \_\_\_\_\_\_barStench?\* **No ☐**\*N.B. The solvent volume, reagent GHS classification, and stench of any reaction components may impact the overall reaction class; refer to Section 6.5 of the Safety Handbook for details. |

**IF YOU CHANGE ANY OF THE CONDITIONS (SCALING UP/DOWN, TESTING DIFFERENT REAGENTS, ALTERING CONCENTRATION ETC.) YOU MUST REVIEW ALL ASPECTS OF THIS HIRAC TO DETERMINE IF ANY HAZARDS OR RISKS ARE ALTERED BY THE CHANGE OF CONDITIONS, AND IF SO, A NEW HIRAC MUST BE COMPLETED.**

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| **RISK CONTROLS** |

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|  |  | **Yes** | **No** |
| **ELIMINATION** | Can this procedure or any of the associated processes be eliminated? | [ ]  | [x]  |
| **SUBSTITUTION** | Can safer alternate processes or less hazardous chemical(s) be used? | [ ]  | [x]  |
| **ISOLATION / ENGINEERING** | Use of a specialist facility (specify): | [ ]  | [x]  |
| Specialist fume cupboard required (e.g. highly toxic, or fume hood with scrubber)? | [ ]  | [x]  |
| Other local Exhaust ventilation | [x]  | [ ]  |
| Is a blast shield required?**\*\*** | [ ]  | [x]  |
| Are minimum volumes for the reaction scale being used? | [x]  | [ ]  |
| Roof closure necessary? | [ ]  | [x]  |
| Special sample storage requirements (specify below): | [ ]  | [x]  |
| Have ignition sources, proximity to other experiments/processes and housekeeping been considered? | [x]  | [ ]  |
| Other | [ ]  | [x]  |
| **ADMINISTRATION** | Additional approvals (after-hours, safety officer etc.) required? | [ ]  | [x]  |
| University Risk Assessment required for any High-Risk Chemicals?\*\*\*  | [ ]  | [x]  |
| Does the workspace have all necessary controls in place to conduct the reaction safely? | [x]  | [ ]  |
| Is additional signage required for your reaction?**\*\*\*\*** | [ ]  | [x]  |
| **PERSONAL PROTECTIVE EQUIPMENT** | Standard PPE required is lab coat, safety glasses, footwear | [x]  | [ ]  |
| Eye / Face protection (specify) | [ ]  | [x]  |
| Gloves | [x]  | [ ]  |
| Hearing | [ ]  | [ ]  |

**\*\*N.B**. A blast shield MUST be used for any reactions involving heating or pressure build-up (e.g. gas evolution) in a sealed vessel. A blast shield must also be used for any reactions involving potentially explosive chemicals (PECs).

**\*\*\*** High-risk chemicals include any scheduled chemicals (e.g. restricted/prohibited carcinogens, scheduled poisons), as well as any other chemicals described in Section 5.8 of the Safety Handbook.
**\*\*\*\*** This includes signs required for the use of scheduled carcinogens, highly toxic reagents, nanoparticles, and PECs, as well as red reaction tags to accompany all High risk and Very High risk procedures. Refer to Sections [5.8](https://au-mynotebook.labarchives.com/share/SAFETY%2520NOTES/NDQuMnw1NjEwMS8zNC9UcmVlTm9kZS8xODcwNDQyMDIyfDExMi4xOTk5OTk5OTk5OTk5OQ%3D%3D) and [5.9](https://au-mynotebook.labarchives.com/share/SAFETY%2520NOTES/NDUuNXw1NjEwMS8zNS9UcmVlTm9kZS8zNDA5MDA5MDE1fDExNS41) for further information.

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| **WASTE DISPOSAL – Consider if your waste is compatible with waste already in the container and allow for pressure build-up prior to sealing** |
| **Waste Type** | **Yes** | **No** | **Waste Type** | **Yes** | **No** | **Waste Type** | **Yes** | **No** |
| Non-halogenated organics |[x] [ ]  Toxic aqueous waste (including heavy metals) |[ ] [ ]  Segregation of waste required (e.g. mercury or restricted chemical waste)? |[ ] [ ]
| Halogenated organics |[ ] [ ]  Solid waste? (Celite/silica) |[ ] [ ]  Other (specify): |[ ] [ ]
| Sharps |[x] [ ]  Acidic/basic aqueous waste?  |[ ] [ ]  Nil waste to drain (DG 9 or reagents with stench)?  |[ ] [ ]

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| **INCIDENT RESPONSE** |
| **EMERGENCY SHUTDOWN** | Turn off any appliances (vacuum, stirrer). Ensure any reagent bottles are closed. |
| **SPILL** | For small spills, wear gloves and use paper towels to sop up liquid. Ventilate area and dispose of paper towels appropriately. For larger spills, alert laboratory safety officer and indicate spill zone to others working in the lab. Ventilate area.  |
| **FIRE/EXPLOSION** | Turn off any appliances and exit building according to instructions of the fire warden.  |
| **CHEMICAL EXPOSURE** | Wash off any chemical residue using eye wash stations and/or safety showers if necessary. Alert first aid officer and seek medical advice if necessary.  |
| **General response for accidental exposure** • Skin/eye contact – wash the affected area with cold running water for 15 minutes (unless otherwise stated in the SDS). Contact a School First Aid Officer• Inhalation – remove to fresh air and consult a School First Aid Officer |

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| **ASSESSMENT OF PROCEDURE CLASS & RESIDUAL RISK** |
| **LOW RISK** | **MEDIUM RISK** | **HIGH RISK** | **VERY HIGH RISK** |
|[ ] [x] [ ] [ ]
| Supervisor approval | Supervisor approval | Supervisor approval andFloor Safety Officer approval | **Delay proposed work****Refer to Safety Committee** |

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| **APPROVAL TO WORK**  |
| **User declaration**I understand the hazards associated with this activity and agree to implement the proposed risk controls. The proposed work [does / does not] involve the use of a scheduled carcinogen or highly toxic chemical (See [Section 5.8](https://au-mynotebook.labarchives.com/share/SAFETY%2520NOTES/NDQuMnw1NjEwMS8zNC9UcmVlTm9kZS8xODcwNDQyMDIyfDExMi4xOTk5OTk5OTk5OTk5OQ%3D%3D) of School Safety Handbook); if I am using such a chemical, I have completed an additional more detailed [University Risk Assessment](https://intranet.sydney.edu.au/science/chemistry/safety-health-wellbeing.html#HIRAC) for handling the scheduled carcinogen(s) or highly toxic chemical(s). |
| Name: | **Klementine Burrell-Sander** | Signature: | **A picture containing arrow  Description automatically generated** | Date: | **19/04/2021** |
| **Supervisor or Delegate Approval**I approved this activity. The proposed risk controls are adequate to manage the associated risk. The user is competent to undertake the reaction and the associated tasks and processes.  |
| Name: |  | Signature: |  | Date: | **27/01/2021** |
| **Floor Safety Officer or Relevant Specialist Officer Approval** (e.g. carcinogen, laser, radiation officer; for high risk activities)I have independently reviewed this risk assessment and approve the work as a delegate of the Head of School. |
| Name: |  | Signature: |  | Date: | **27/01/2021** |

**Risk Matrix**

