**Reaction Risk Assessment Form Reaction Number: 1\_SC\_Tues\_22**

Below give a scheme for any reaction being carried out and write the full procedure including work-up, purification methods and full method for any analysis of qualitative tests to be carried out (*e.g.* chromatography, TLC, NMR, IR or test tube reactions). You must also give details of the procedure for any preparation of solutions you will need to carry out for the experiment (e.g. acid/base or TLC stain solutions, cold baths). Use the form below to assess the risks associated with **ALL** the reaction and process conditions (*e.g.* heating, cooling, vacuum), particular hazards (*e.g.* exotherm, gas evolution, flooding, asphyxiation, burns—hot or cold, explosion, needle stick injuries), quench procedures and waste disposal as well as the chemicals to be used, including your expected product, solvents and known by-products.

**Reaction details**





**Procedure**

D-Sorbitol (4.90 g) was weighed into a 3- necked round-bottom flask fitted with Dean-Stark apparatus. Cyclohexane (35 ml) and methanol (10 ml) were added, and the mixture was stirred under N2 at 50°C for 20 min. Methyl 4-formylbenzoate (7.50 g) and p-toluene sulfonic acid hydrate (1.00 g) were dissolved in methanol (20 ml) and stirred for 20 min at room temperature, before being added dropwise to the D-sorbitol mixture. The reaction temperature was increased to 70°C, and was stirred for 2 h, until most of the solvent was removed. The white paste formed was washed with methanol (3 x 100 ml). The crude product was dried under high vacuum for 2 h, then air-dried overnight. The product was washed with boiling water (4 x 100 ml) and boiling toluene (3 x 100 ml) respectively.

| Complete the following table for all reagents, solvents and materials used in the experiment (e.g. including drying agents, chromatography stationary phase etc.). | | | | | | **Chemical hazards and routes of exposure** | | | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Route of exposure: 1, Inhalation; 2, Skin/eye contact; 3, Swallowing | Carcinogen, teratogen, mutagen | Very toxic/toxic | Harmful/irritant | Explosive | Pyrophoric | Highly flammable/flammable | Oxidising | Corrosive | Lachrymator | Other (specify): |
| **Compound** | **FW** | **d (g/mL)** | **Quantity (g or ml)** | **mmols** | **Molar Equiv** |  | **1,2,3 for these columns** | | | **Use X for these columns** | | | | | |  |
| D-sorbitol | 182.17 |  | 4.90 g |  |  |  |  |  |  |  |  |  |  |  |  |  |
| Cyclohexane | 84.16 | 0.779 | 35 ml |  |  |  |  | X | 2 |  |  | X |  |  |  |  |
| Methanol | 32.04 | 0.79 | 330 ml |  |  |  |  | 1 2 3 |  |  |  | X |  |  |  |  |
| Methyl 4-formylbenzoate | 164.16 |  | 7.50 g |  |  |  |  | X |  |  |  |  |  |  |  |  |
| Toluene 4-sulfonic acid | 190.22 |  |  |  |  |  |  |  | 1 3 |  |  |  |  |  |  |  |
| Toluene | 92.14 | 0.865 | 300 ml |  |  |  |  |  | 2 |  |  | X |  |  |  | Harmful to aquatic life. |
| DBS-CO2Me (Product) | 474.46 |  |  |  |  |  |  |  |  |  |  |  |  |  |  | Unknown, treat as hazardous |
| TBS-CO­2Me  (Side Product) | 620.6 |  |  |  |  |  |  |  |  |  |  |  |  |  |  | Unknown, treat as hazardous |
| MBS-CO­2Me  (Side Product) | 328.32 |  |  |  |  |  |  |  |  |  |  |  |  |  |  | Unknown, treat as hazardous |

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|  | **Repeat Experiment?** (This lab project only) **Previous assessment Reaction Number:** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **New Experiment:** Supervision Category (please tick one) | | | | | | | | | | | | | **A** ✓ | | | | | | | | | | **B** | |  | | | **C** | | | **D** |
|  | **Standard protocol followed?** (Please give a reference here if you are following/closely adapting a known literature method) | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|  | **Reaction conditions/processes and associated hazards**  Complete the table below with all reaction conditions (e.g. heating, cooling, vacuum) and associated physical hazards (e.g. exotherm, gas evolution, flooding, asphyxiation, burns—hot or cold, explosion, needle stick injuries). | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Reaction condition or process** | | | | | | | | | | |  | | | | | | | **Physical hazards associated** | | | | | | | | | | | | | |
| Fumehood | | | | | | | | | | |  | | | | | | | Asphyxiation | | | | | | | | | | | | | |
| Dean-Stark Apparatus | | | | | | | | | | |  | | | | | | |  | | | | | | | | | | | | | |
| Vacuum desiccator | | | | | | | | | | |  | | | | | | |  | | | | | | | | | | | | | |
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| **Control Measures**  (Please tick boxes) | | | Safety glasses ✓ | | | | | | Lab coat ✓ | | | | | | | | Fume hood ✓ | | | | |  | | | | | Safety Screen | | | | |
|  | | | | Gloves (type): Nitrile ✓ Marigolds Red gloves Other | | | | | | | | | | | | | | | | | | | | | | | | |
| Scrubbing train (type): | | | | | | | | | | | | | | | |  | | | | | Other: | | | | | | | |
|  | **Emergency procedures**  Are there any specific emergency procedures (e.g. first aid, decontamination or firefighting measures) necessary for this process? Add in emergency PXXX hazards from the SDSs used here.  Cyclohexane  IF SWALLOWED: Immediately call a POISON CENTER/ doctor. Do NOT induce vomiting.  IF ON SKIN (or hair): Take off immediately all contaminated  clothing. Rinse skin with water.  Methanol  IF SWALLOWED: Immediately call a POISON CENTER/ doctor.  IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water.  IF INHALED: Remove person to fresh air and keep comfortable for breathing. Call a POISON CENTER/doctor.  Toluene 4-Sulfonic acid  IF ON SKIN: Wash with plenty of water.  P305 + P351 + P338 IF IN EYES: Rinse cautiously with water for several minutes.  Remove contact lenses, if present and easy to do. Continue  rinsing.  Toluene  IF SWALLOWED: Immediately call a POISON CENTER/ doctor. Do NOT induce vomiting.  P303 + P361 + P353 IF ON SKIN (or hair): Take off immediately all contaminated  clothing. Rinse skin with water. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|  | **Reaction and/or reagent quench**  (Give quench reagent and procedure and any possible hazards): | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|  | **Waste disposal**  Complete the table below to outline how all waste chemicals from this experiment will be disposed of (including reaction products). If special precautions are required (e.g. quenching before disposal) please describe details under ‘other’. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **List All reagents, products, and quench materials** | | Chlorinated waste | | | Non-chlorinated waste | | | | | Silica waste | | | | | | Aqueous  Metal waste | | | | | | | Clinical waste | | | | | | Other/details | | |
| D-sorbitol | |  | | |  | | | | |  | | | | | |  | | | | | | | X | | | | | |  | | |
| Cyclohexane | |  | | | X | | | | |  | | | | | |  | | | | | | |  | | | | | |  | | |
| Methanol | |  | | | X | | | | |  | | | | | |  | | | | | | |  | | | | | |  | | |
| Methyl 4-formylbenzoate | |  | | | X | | | | |  | | | | | |  | | | | | | |  | | | | | |  | | |
| Toluene 4-sulfonic acid | |  | | | X | | | | |  | | | | | |  | | | | | | |  | | | | | |  | | |
| Toluene | |  | | | X | | | | |  | | | | | |  | | | | | | |  | | | | | |  | | |
| **Overall risk rating** (select one rating) | |  | | | | **Risk Assessment Matrix** | | | | | | | | | | | | | | | | | | | | | | | | | |
| **SEVERITY** | | Fatality | | | | Medium | | | | High | | | | | | | | High | | | | | | High | | | | Unacceptable | |
| RIDDOR | | | | Medium | | | | Medium | | | | | | | | High | | | | | | High | | | | High | |
| Moderate injury | | | | Low | | | | Low | | | | | | | | Medium | | | | | | Medium | | | | Medium | |
| Minor injury | | | | Insignificant | | | | Low | | | | | | | | Low | | | | | | Low | | | | Low | |
|  | | | | | | Unlikely | | | | Possible | | | | | | | | Probable | | | | | | Probable | | | | Certain | |
|  | | | | | | **LIKELIHOOD** | | | | | | | | | | | | | | | | | |
| **Justification for rating** (describe reasoning for risk rating) | |  | | | |  | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Members of group:**  All members of the group must read and understand this risk assessment (including any comments from the lab supervisor/demonstrator given below) then sign to confirm you have done this before starting work in the lab. **You must not make any changes to the procedure stated without prior agreement with the supervisor which should be indicated in the comments box below**.  Signature:………………………………Name:………..……………………  Signature:………………………………Name:………..……………………  Signature:………………………………Name:………..……………………  Signature:………………………………Name:………..……………………  **Date:** | | | | | | | | | | | | | | |  | | | | | | **Designated supervisor signature:**  **Date:** | | | | | | | | | | |
|  | **Comments from lab supervisor** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|  | If you receive comments, then please address them when writing your new risk assessment.  Mostly likely you will need a new risk assessment each week/every 10 hours of lab time.  Staff will not countersign a risk assessment that is covered with additions and amendments as this is not easy to refer to in an emergency. This will result in a yellow card as it being an unsafe lab practice. | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |