University of St Andrews
Scotland's first university

Example RA #1 - Methylation of Phenol Derivatives

School of Chemistry

Not submitted

Supervisor:
Creator:

Workers directly involved in the procedure DRAFT

#	User	Signed
1		

Locations for the procedure DRAFT

#	Building	Rooms
	Purdie Building	

Substances to be used in the procedure DRAFT

#	Name	Hazard rating	STEL	LTEL	Licence
1	sodium hydride	4 F X			
2	acetone	2 F	1500 ppm or 3620 mg/m3	500 ppm or 1210 mg/m3	
3	tetrahydrofuran	3 F T	100 ppm or 300 mg/m3	50 ppm or 150 mg/m3	
4	ethyl acetate	2 F	400 ppm or 1468 mg/m3	200 ppm or 734 mg/m3	
5	hexane n-	4 F T		20 ppm or 72 mg/m3	
6	silica gel fine (various types)	4 A		6 mg/m3	
7	iodomethane	5 C T		2 ppm or 12 mg/m3	

Hazards and control measures associated with experimental procedure.

Example 1 - Chemical Hazards
To a solution of 4-fluorophenylacetic acid (toxic; avoid direct contact - wear nitrile gloves when handling) and iodomethane (toxic, carcinogen; avoid direct contact - wear two pairs of nitrile gloves when handling; handle exclusively within a fume cupboard) in THF (flammable; avoid sources of ignition) was added NaH (flammable, explosive; avoid direct contact, sources of ignition and exclude moisture) portion-wise at 0 C. The product was purified by silica gel column chromatography (silica dust is a respiratory irritant; avoid inhalation of dust by handling within fume cupboard) using hexane and ethyl acetate (flammable; avoid sources of ignition).

Example 2 - Process Hazards
Ferric nitrate (causes severe skin burns and eye damage - wear protective gloves when handling) and barium nitrate (toxic by ingestion, harmful by inhalation, oxidiser - keep away from ignition sources - wear protective gloves when handling) are placed into a crucible and placed into a furnace at 1000 degrees celsius (risk of severe burns if crucible / material at high temperature makes contact with skin; Heat resistant gloves, crucible tongs and protective face shield with darkened lenses will be used when adding / removing the sample from the furnace. Hot samples will be left on heat resistant mats to cool before further processing).

Example 3 - Biological Hazards
The main hazard comes from any pathogens (parasite or virus e.g. Hepatitis B/C) that may be present in the samples. Blood samples and sera (prior to inactivation) must be handled in a Class II microbiological safety cabinet to reduce the risk of exposure to aerosols. Centrifuging should be done in buckets fitted with Biosafety lids. Virus inactivation may be via heat treatment (56?C for 3 minutes) or treatment with 1% Triton X-100 . Labcoat, gloves, and eye protection must be worn. Contaminated tips, tubes, and disposable plasticware will be sterilised by autoclaving. Liquid waste will be disinfected using Virkon 1% for 1 hr.

DRAFT

Safety considerations associated with experimental scale.

Examples of Approaches to Discuss Experimental Scale:

* Highlight maximum scale before risk assessment is reviewed
Experiment will be conducted up to 10 mmol scale with regard to the starting acetic acid (<1 g acid). Experiments >10 mmol will be reassessed to ensure control measures are sufficient.

* Example from literature:
Procedure adapted from "journal reference" (A.Nother) where it was conducted on a 20 mmol scale. Planned reaction will be conducted on 10 mmol scale and does not pose any significant change in risk.

* Increasing reaction scale:
Experimental procedure based on literature precedent conducted on 1 g scale of starting acid. Experiment will ultimately be performed on 20 g of acid, however, reaction will be scaled step-wise to determine if the exotherm generated is suitably controlled by experimental set-up or if additional cooling is required.

This experiment does not require Health Surveillance.

Other workers in the area
- Cleaning Staff
- Other researchers

Notification method
- Notice will be displayed on the door
- Notice will be displayed on the experimental bench
- Hazards will be discussed with the workers

Risks of Process
- Burns
- Fire
- Toxic

Route to risks
- Direct contact
- Inhalation

Engineering Control measures
- Fume cupboard

Personal protective equipment
- Eye protection
- Hand protection
- Laboratory coat

Other control measures DRAFT

Methyl iodide is a volatile, carcinogenic material. The substance will be handled exclusively within a fume cupboard and two pairs of gloves will be worn to protect against direct contact with skin.

The reaction involves heating a peroxide/organic azide/diazonium salt, which poses a potential explosion risk. The reaction vessel will be placed behind a blast shield throughout the duration of the experiment.

Experiment will use highly toxic carbon monoxide gas. Fixed and personal gas detectors will be used to monitor CO levels.

This experiment requires general training

This experiment requires monitoring

Waste

Solid waste
- Low level contaminated waste

Organic liquid waste
- Solvent waste collection
- Treat organic solvents containing methyl iodide with NaOH before disposal

Aqueous liquid waste
- Wash to drain
- Keep for special waste disposal

Special procedures require to make waste safe prior to disposal DRAFT

* Organic solvents and aqueous solutions treated with NaOH solution to destroy trace amounts of methyl iodide
* Needles used to dispense methyl iodide treated with NaOH solution before disposal
* All glassware that has come into contact with methyl iodide will be washed with NaOH solution.

Emergency

Fire
- Fire blanket
- CO2 fire extinguisher
- Call fire service

Burns
- Irrigate with water
- Call first aider

Chemical spillage
- Clean up
- Experiment involves the use of liquid bromine. Have 25% sodium thiosulfate solution on hand to neutralise minor spills.