Task Risk Assessment
With COSHH Compliance

Before Controls	After Controls
HIGH 20 RISK	LOW 5 RISK

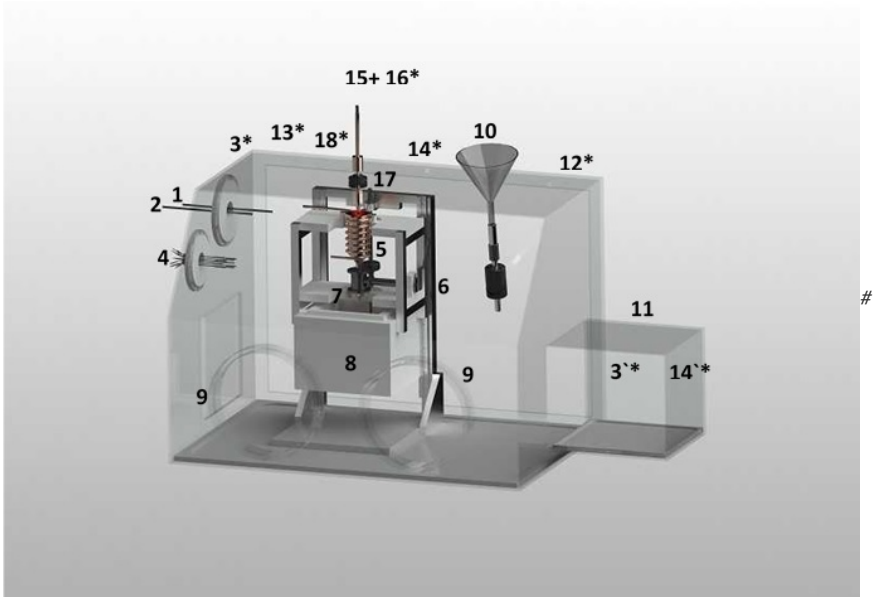
Task Title	Generating Icy Particles in Cryo-Ethane		
Process Title	(1) Start-up, (2) Reaction vessel cooling, (3) Ethane liquifaction, (4) Making icy particles, (5) Sample recovery		
Location	Stem Faculty		
Assessed On	16/01/2020	**Assessed By**	Thomas Webley, Vincent Deguin, Peter Landsberg, Helen Fraser
Review Date	16/01/2021	**Reference**	RA816548
Signed By	Thomas Webley	**Revision**	4
People Affected	Employees, Visitors, Lab Users		

Hazards

PPE

Task/Procedure/Method Statement

Procedure



 # CAD drawing of experimental apparatus. Numbers will be referred to throughout assessment.

(1) Start-up

• Add all the materials which need to go inside GB:
  - o Frost shield
  - o Q-tip (Clear condensation/freezing at the end of nozzle)
  - o Paper sheet (monitor water flux within GB)
  - o Towel
  - o Tweezers (*2) to handle cold materials
  - o Cryo-Gloves

• Turn on computer

• Turn on Lakeshore PID, Eurotherm and check Input parameters are correct

• Check placement, integrity and functionality of thermocouples

• Check and record all experimental base parameter

• Close the port at the rear of the glove box (2 persons needed)

• Screw firmly in place using screw driver

• Turn on N2 flow to main glove box [14] (0.2 Bar)

• Allow time for nitrogen flow to remove air from glove box (60 mins). (N.B., it has been experimentally verified that this is a sufficient amount of time to allow oxygen to be fully purged from the glove box).

• Reduce N2 flow below 0.1 Bar to maintain a slight overpressure and a comfortable working environment.

• Ensure that pressure reading on gauge [13] is 0.

(2) Reaction vessel cooling

• Check that pipe outlet [2] is inserted in a Dewar.

• Open Labview -> “Program name”

• Start Labview Temperature recording program (time = 0)

• at time = 1 min, open the 'liquid fill/decant' valve on liquid nitrogen vessel attached to [1] to begin coolant flow (flowing through 1 -> 5 -> 2)

• When copper vessel [5] has cooled (should take <10 min) to base temperature, activate PID controller attached to heater on this vessel and set temperature to desired range. Allow a few minutes for this value to stabilize.

• Ensure that the cooling matches reference curve before proceeding to the next process.

(3) Ethane liquifaction

• Clean ethane line by purging with a small amount of ethane gas. Open the valve slightly open, then close in this order: ethane bottle, regulator, [12].

• Position ethane line (tubing in glove box from [12]) over copper vessel [5]. Make sure that pipe is 1 cm deep inside reaction vessel and that it is not in contact with the copper wall.

• Move Dewar up and down to adjust pipe position.

• Open valve at top of ethane cylinder.

• Slowly increase the pressure on the regulator`s outlet to 80 mbar.

• Open ethane inlet valve [12] and observe ethane liquefaction.

• Heater temperature at copper vessel [5] can be adjusted using PID controller attached to [4] to encourage ethane liquefaction

• When enough liquid ethane formed (i.e., when liquid ethane level is 2cm from the top -> 230 cm3), close the valve at the top of the ethane cylinder and drain out volume between bottle and regulator to stop flow.

• Wait a few seconds and then close ethane inlet valve [12].

• Move the ethane tube line from [12] well away from the copper vessel [5].

(4) Making icy particles

• Fill nebulizer water reservoir with deionized water or D2O.

• Check temperatures:
  - o End of nozzle temperature must be stable and above 0 °C
  - o Nozzle heater block must be below 50 °C

• Slowly introduce nitrogen pressure into the nebulizer [15] and [16] at (0.5 bar).

• Turn on Nebulizer.

• Wait few seconds (necessary to start the nebulization), then control the quality of the flux (a mist should be visible).

• Once OK, level up the Dewar to reach an equilibrium between T of the nozzle and efficiency of the water droplet insertion (1-3 cm from ethane surface).

• Remove plug on top of nozzle.

• Connect water reservoir and copper nozzle (start timer).

• Water ice production time (20 min).

• During introduction, check that no frost is forming on the nozzle. If ice does form, stop the introduction by disconnecting water reservoir and nozzle, put the plug in place and clean nozzle mouth with Q tip if necessary.

• Control:
  - o Quality/speed of water flow to ensure that there is no condensation or freezing at the end of pipe.
  - o Level of water in nebulizer reservoir.
  - o Level of liquid ethane.

• When enough sample produced (~20 min introduction), disconnect water reservoir from nozzle.

• Replace plug on top of nozzle.

• Turn off nebulizer and shut N2 gas valve.

Substance Information

Product Name
DEUTERIUM OXIDE D 99 9 (DLM-4)

Manufacturer
Cambridge Isotope Laboratories

Hazard Symbols


Application Method	Application Area	Frequency Of Use	Quantity Used	Duration Use
flowed through nebuliser	nebuliser and in samples at end	In regular use	few litres	Several hours

Spillage, Storage & Waste

Spillage; prevent further leakage or spillage if safe to do so. Do not let product enter drains. Wipe up with absorbent material (e.g. fleece, cloth). SDS also references respiratory protection, so consider if this is appropriate for handling of the spillage. Hazards may arise from contact with incompatible materials or electrical equipment. Keep spill away from any such equipment or exposed wires, turn off power to these systems if necessary.

Storage; keep container closed in a dry and appropriately-ventilated place, store at room temperature away from light and moisture.

Disposal; materials used to wipe up a small spillage may be disposed of in laboratory waste. Disposal needs meet local regulations.

WELS

None quoted on SDS

Product Name
WATER (H20)

Manufacturer
The Almighty

Hazard Symbols


Application Method	Application Area	Frequency Of Use	Quantity Used	Duration Use
flowed through nebuliser	nebuliser and ices	in regular use	few litres	several hours

Spillage, Storage & Waste

Spillage; mop up any spills. Hazards only likely to arise from combination with incompatible materials or electrical equipment. Keep spill away from any such equipment or exposed wires, turn off power to these systems if necessary.

Storage; where high purities are required, leave sealed to maintain purity level. Avoid incompatible materials (check SDS for other compounds in surrounding environment).

Spillage; can be disposed of via sink or drain. Materials used to mop up spill can be considered non-hazardous and disposed of in lab bin unless they are also contaminated with other compounds.

WELS

N/A

Product Name	Manufacturer	Hazard Symbols
Ethane (00582)	Sigma-Aldrich Company Ltd	

Regulatory Statements

H220: Extremely flammable gas

H280: Contains gas under pressure; may explode if heated

P210: Keep away from heat/sparks/open flames/hot surfaces - No smoking

Application Method	Application Area	Frequency Of Use	Quantity Used	Duration Use
to make a cryoliqiud	dewar and flow in tubes	all the time when making cryoethane	small flow from cylinder	up to hours during cryoethane production

Spillage, Storage & Waste

Spillage: do not allow to accumulate where air or oxygen is present. Turn of valves immediately if leak is suspected. Excess flow into chamber can be pumped to trap in line then released slowly, at a later time. Do not pump large amounts of ethane directly to atmosphere. Always evacuate area and raise alarm if significant concentrations of ethane are thought to have reached air. Do not re-enter lab until these have dispersed.

Storage: keep away from sources of ignition, direct sunlight, sparks or flames. Where possible, store outside of laboratory and in suitable - well ventilated - flammable gas storage area. Always close all valves and leave sealed in between usages. Cylinder must be secured in a stable manner to prohibit any slip or fall.

Waste: all used ethane should exhaust to the in-line carbon trap where it will be adsorbed. The trap contents should be disposed of as chemical waste when approaching capacity; do not attempt to refresh and be aware that the effective flow rate can be litres per minute.

WELS

None quoted in SDS

Product Name	Manufacturer	Hazard Symbols
Nitrogen (300000000099)	Air Products Ltd	

Regulatory Statements

H280: Contains gas under pressure; may explode if heated

P403: Store in a well ventilated place

Application Method	Application Area	Frequency Of Use	Quantity Used	Duration Use
flowing to get inert atmosphere	two glove boxes	regular use	difficult to quantify; many multiple litres [limited by cylinder capacity]	several hours

Spillage, Storage & Waste

Note; controls relate to use of gas lines where cylinder is stored externally. Additional controls apply for safe use of cylinders.

SPILLAGE: for large spillage evacuate the area, especially if low O2 alarm sounds, do not re-enter the area unless atmosphere is proved to be safe. Ensure adequate air ventilation. Prevent from entering sewers or any place where its accumulation can be dangerous. Do not allow large spillages to escape into unventilated corridors.

STORAGE: all valves should be closed when not in use. Small volumes can be left in the line after a closed valve, there is no need to evacuate these. Gas lines must be proven to be secure.

WASTE: waste gaseous nitrogen can be allowed to exhaust to atmosphere.

WELS

N/A

Product Name	Manufacturer	Hazard Symbols
Nitrogen, refrigerated liquid (118187)	Linde Gas Hungary Kft.	

Regulatory Statements

H281: Contains refrigerated gas; may cause cryogenic burns or injury

P282: Wear cold insulating gloves/face shield/eye protection

P336: Thaw frosted parts with lukewarm water. Do not rub affected areas

P403: Store in a well ventilated place

Application Method	Application Area	Frequency Of Use	Quantity Used	Duration Use
in making icy particles	dewars, decanted and used inside glove box	regular use	litres	several hours

Spillage, Storage & Waste

SPILLAGE: for large spillage evacuate the area, especially if low O2 alarm sounds, do not re-enter the area unless atmosphere is proved to be safe. Ensure adequate air ventilation. Prevent from entering sewers or any place where its accumulation can be dangerous. Liquid spillages can cause weakening of, or damage to, structural materials.

STORAGE: moving dewars, even for short distances, use a cart/dewar trolley. Ensure dewar caps in place until the container is stable in the lab. Dewars should be checked for damage or signs of leakage before use. Dewars should be stored away from combustible materials, heat or sources of ignition. Liquid nitrogen should only be stored in the lab fitted with, or containing, a low O2 alarm.

WASTE: waste liquid nitrogen can be allowed to boil off in a fume hood or similar extraction direct to external atmosphere.

Additional handling and storage controls apply to pressurised dewars.

WELS

N/A

Task Related Hazards & Controls

Before Controls: **Medium Risk** (10/25)

REQUIRED CONTROLS

After Controls: **Low Risk** (5/25)

Hazard Description

Ethane cylinder: Contains gas under pressure; may explode if heated

Controls In Place


Store away from all sources of combustion in suitable containment for a flammable material.


Gas cylinder must be left secured to avoid any movement or falls which could put strain on attached gas lines.


Cylinders must be protected from excessive heat and/or sparks. No open flame; Fire, open ignition source and smoking prohibited.


Ensure that gas cylinder is fitted with appropriate, in-date regulator and flashback arrestor. This must be installed and leak-checked by a suitably trained individual.


Store cylinders outside of laboratories wherever possible. Where this is impossible, ventilation must be considered with regard paid to the fact that ethane gas can form explosive mixtures with air at levels as low as 2.9% concentration.

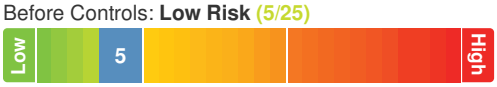
Emergency Controls In Place


Raise alarm if fault suspected with any cylinder storing potentially flammable or explosive gas. Report that an ethane cylinder may be present in the case of e.g. fire.


If safe to do so, close all valves to isolate cylinder and lines in case of emergency.


If a fault is suspected with a pressurized cylinder, evacuate area after closing valves.

Before Controls: **Low Risk** (5/25)



REQUIRED CONTROLS

After Controls: **Low Risk** (5/25)

Hazard Description

Risk of asphyxiation from lowered oxygen environment when using gases. Liquified gases expand hugely on boiling.

Controls In Place

Switch off gas flows when not use. In case of emergency, switch off if safe to do so.

Set-up is designed to ensure that only gaseous nitrogen will be released from the coolant stream to exhaust.

Where possible, ensure that the total volume of gas resulting from liquefied gases boiling is lower than the volume require to drop the air-oxygen content below 19.5%. Where this is not possible, additional extraction should be considered.

Unlike initial iterations of this set-up, the current procedure is now conducted in a lab with a low level fan that will automatically trigger if low levels of oxygen are detected. This does not mitigate the need to leave the lab if a low oxygen alarm is triggered.

Gas flows must be taken to local extraction. Ensure that outflow from these is not able to build-up within the lab.

Liquid nitrogen may be used for cooling in large volumes. This should be released in gaseous form to exhaust but large volumes of cold gas may be generated. This must vent to a suitable location, either a separate connection to extraction or direct to atmosphere, although this must not be at a height or location which could affect anyone passing the building. If extracting to a local exhaust system, monitor this for signs of ice build-up which could limit flow.

Emergency Controls In Place

Do not enter any lab where a low-oxygen alarm is sounding without appropriate respirator (this is only likely to apply in case of professional rescue service).

Always ensure that low-oxygen alarm is present, and working, in any space when using extended gas flows or liquefied gases. Evacuate immediately if alarm sounds.

Before Controls: **High Risk** (20/25)

Low 20 High

REQUIRED CONTROLS

After Controls: **Low Risk** (4/25)

Low 4 High

Hazard Description

Ethane: Extremely flammable gas

Controls In Place

Check all gas lines are secure before starting any work involving them.

Switch off gas at cylinder and close other available valves whenever not in use.

Flammable gases and gas lines must be protected from excessive heat and/or
sparks. No open flame; Fire, open ignition source and
smoking prohibited.

Ethane must not be allowed to flow into
environments with potentially raised temperatures, unless
a specific assessment exists to cover this risk as part of a
laboratory procedure.

Allow any flammable gas within the lines to flow away with excess nitrogen gas after use. NEVER store ethane in the gas lines used in this set-up for more than the duration of the experiment.

All installation, maintenance work and alterations to lines must be performed by an individual with sufficient training for pressurized gas line manipulations. Changes should not be made by any unqualified individual.

Before the first instance of using the glove box, nitrogen will be flowed through whilst an oxygen gas detector is present in the chamber. It should be proven that the procedure will lead to a low-oxygen environment, including a margin of error.

Do not begin ethane flow into glove box until it has been purged of air with nitrogen. Maintain nitrogen flow throughout experiment and ensure that this remains the major constituent of the atmosphere inside the glove box throughout the experiment.

The set-up has been adjusted to cope with any expected risks of the procedure and incorporates thermal barriers (PTFE feedthroughs), flexible tubing where liquid nitrogen feed connects to copper vessel, dedicated raising/lowering system of the reaction vessel to keep it at the correct height etc. If there is a need to replace any part in the system, ensure that it is replaced with an equivalent part.

A previous iteration of this experiment incorporated an in-line carbon trap to remove any excess ethane from the exhaust flow. The set-up was found to be exhausting ethane in only low concentrations, highly diluted with nitrogen and with the ethane liquifying extremely quickly within the reaction vessel. The trap was also found to be adding resistance to the exhaust line. This was therefore removed as an unnecessary precaution with an operational impact.

Emergency Controls In Place

In case of emergency, including liquid nitrogen spillage on the floor of the glovebox whilst ethane is present inside, evacuate area.

If safe to do so, close all valves to isolate cylinder and lines in case of emergency. Do not attempt if this involves increased risk of harm to user or others.

If any flammable gas leak is
suspected, raise the alarm
immediately.

Experimental setup assumes that all gas will be flowing into an oxygen free (or greatly reduced) environment. Treat any signs of ethane escaping this environment as an emergency and apply controls listed here.

Before Controls: **Medium Risk** (12/25)

Low 12 High

REQUIRED CONTROLS

After Controls: **Low Risk** (2/25)

Low 2 High

Hazard Description

Cryogenic or gas; may cause cryogenic burns or injury

Controls In Place

Do not touch with naked skin.

Specific cryogenic gloves must be used when handling ultra-cold materials.

Cryogenics should only be handled by a trained individual, or with a trained individual supervising, who understands hazards, PPE, etc.

When refilling Dewars through the funnel attachment on the glovebox, liquid nitrogen must be decanted slowly to ensure no overflow or spillage.

Wear a labcoat when handling cryogenic liquids. For manipulating larger volumes or when decanting at an elevated height, a cryo-apron may be necessary for extra protection.

Wear suitable eye protection. Safety glasses may be appropriate for some elements of the work but a full-face shield should be used when manipulating larger volumes or when decanting liquid nitrogen at an elevated height (i.e. into the funnel attachment on the glovebox).

Emergency Controls In Place

In case of cold burn, flush the affected area with lukewarm water [do NOT use hot water] and contact a medical professional.

Before Controls: **Medium Risk** (10/25)

Low 10 High

REQUIRED CONTROLS

After Controls: **Low Risk** (2/25)

Low 2 High

Hazard Description

Risk of electrical equipment sparking in atmosphere containing explosive gas/vapours (ethane)

Controls In Place

Switch off all electrical equipment when not in use.

Ensure that all electrical equipment and copper gas lines running into the glove box are properly earthed before use.

Inspect all electrical equipment is tested and functional before use. Do not use where there are any signs of damage.

Do not use electrical components inside the glove in the presence of ethane where there is risk of oxygen present in significant quantities in the glove box (i.e. only whilst the internal atmosphere has been purged with nitrogen).

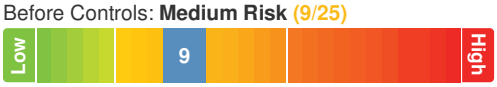
Advice has been sought during set-up regarding parts such as electrical feedthroughs for thermocouples to ensure they are suitable, including at low temperatures. If these need to be replaced it should be with like-for-like parts. Ask for advice if unsure.

System set-up will be designed to minimize any risk of electrical components sparking or glowing inside the glove box. Electric components will be started before the ethane flow, wherever possible, and always after the glovebox has been flushed through with nitrogen to remove oxygen within. They must be turned off before nitrogen flow is stopped.

Emergency Controls In Place

In case of electrical equipment failure or fire, switch off ethane flow immediately but leave nitrogen flow running into the glove box if ethane is though to be present - if it is safe to do so. Evacuate and raise alarm if any danger remains.

Before Controls: **Medium Risk** (9/25)



REQUIRED CONTROLS

After Controls: **Low Risk** (2/25)

Hazard Description

A heater is being used to prevent freezing which could lead to a hot surface outside the glove box

Controls In Place

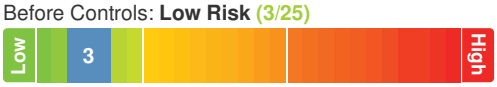
Switch off after use and allow to cool.

Thermocouples will be measuring the temperatures at the nozzle and the outer copper pipe by the heater. The temperature at the heater will be limited to 60 degrees centigrade.

Emergency Controls In Place

If any burn from a hot surface occurs, run affected area under cold water for at least 15 minutes and seek first aid.

Before Controls: **Low Risk** (3/25)



REQUIRED CONTROLS

After Controls: **Low Risk** (2/25)

Hazard Description

Set-up includes both nitrogen and ethane cylinders present in lab.

Controls In Place

Ensure controls listed above relating to securing gas cylinders, in-date regulators, ensuring that gas lines are secure and maintenance is performed by a trained individual, switching off valves and gases after use, and checking lines before use are familiar and applied to all gas cylinders in use.

Emergency Controls In Place

Additional gas volumes in the lab can increase the risk of the asphyxiation hazard. Make sure that controls listed above relating to use of a low oxygen alarm and evacuation if this triggers, as well as switching off all active gas lines in case of emergency (where safe to do so) are familiar and considered.

Before Controls: **Low Risk** (3/25)

REQUIRED CONTROLS

After Controls: **Low Risk** (1/25)

Hazard Description

Solid ethane could be formed if liquid splashes into liquid nitrogen.

Controls In Place

Experiment is designed to limit possibility of splashing of cryo-ethane once formed.

All liquids in system should be fully boiled away before opening glove box or stopping nitrogen flow. This would allow any solid ethane to melt and then boil.

General Controls & Emergency Controls

Controls In Place


Wear eye protection, safety glasses are sufficient for most parts of this procedure however dispensing large volumes of liquid nitrogen, or pouring from an open Dewar, will require a facemask.


Secure gas cylinders


Use extraction to remove gas flows through glove box.


Wear lone worker alarm if necessary


Wear protective gloves - ensure that any thermally protective gloves being used to handle liquid nitrogen are rated for protection at those temperatures.


No open flame; Fire, open ignition source and smoking prohibited


Information - ensure notice on door stating ethane compressed gas/dangerous substance in use. For any work before 9am or after 5pm or when contractors are present, ensure security informed when work starts and stops, +53666


Do not touch any cooled areas or cold substances without protective gloves.

Emergency Controls In Place


If necessary to call for assistance, this should be done away from any dangerous areas.


Contact in case of serious injury to person(s), e.g. cold burns.


Sound the alarm in case of fire or dangerous chemical escape.


Evacuate to assembly point should any alarm trigger


Do not extinguish with water


Sound Alarm - if O2 alarm sounds evacuate

Health Surveillance

No substances in use which are likely to give rise to long-term symptoms.