# Task Risk Assessment
With COSHH Compliance

| Before Controls | After Controls |
|---|---|
| MEDIUM 10 RISK | LOW 5 RISK |

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|---|---|
| **Task Title** | FTIR spectroscopy of solid ice films of pure, layered and mixtures of ethane and water |
| **Process Title** | Preparation and vapour deposition of ethane and water on to a pre-cooled (20 K) ZnSe substrate for in situ FTIR spectroscopy using the UHV Portable Astrochemistry Chamber in Perry D, Lab 016 |
| **Location** | Stem Faculty |
| **Assessed On** | 03/09/2020 |
| **Assessed By** | Anita Dawes, Vincent Deguin, Lily Hills and Tom Webley |
| **Review Date** | 03/09/2021 |
| **Reference** | RA997849 |
| **Signed By** | Anita Dawes |
| **Revision** | 0 |
| **People Affected** | Operators, Visitors, Lab Users |

Hazards

PPE



## Task/Procedure/Method Statement

The procedure detailed here is for the use of the Portable Astrochemistry Chamber (PAC) in Perry D, Lab 016 to measure in situ infrared spectra of vapour deposited solid films of pure, layers and mixtures of ethane and water at temperatures between 20 and 200 K

**Portable Astrochemistry Chamber (PAC) Overview:**

For the general procedure for setting up the PAC vacuum system, please refer to **RA839550**. Briefly, the procedure involves:

1. Pumping down PAC (to $10^{-9}$ mbar at room temp.) via turbo backed by a rotary pump
2. Cryo-cooling a ZnSe substrate – connected to the cold finger of a closed cycle He cryostat
3. Optimising the FTIR spectrometer – aligning optics, cooling the detector (using LN2) and purging the beam path (with a flow of dry compressed air)
4. Liquid vapour/gas sample preparation (water and ethane) and mixing using a dedicated gas line on PAC
5. Vapour deposition onto a pre-cooled substrate and monitoring thin film growth using a He-Ne laser and a photodiode
6. Rotation of the sample, via UHV rotary feed-through and acquisition of IR spectra
7. Thermal processing of ice films using a PID temperature controller
8. Electron irradiation (low energy and contained, to not represent concern outside the chamber)
9. Sample annealing and cleaning (in situ) using the temperature controller
10. Occasional venting of the vacuum system may be required to remove and clean/replace the substrate

PAC is fitted with a gas line consisting of CF, KF and Swagelok vacuum fittings, a gas mixing volume and a dedicated pumping system connected to a rotary pump which can be isolated with an on/off valve. The gas line is isolated from the PAC via an all-metal needle valve and an on/off valve. The gas line has provision to for up to three gas/liquid samples via Swagelok connections, each individually isolated with an on/off valve and a needle valve for controlled filling of the gas line. In this procedure the following connections are made to the gas line:

- pressurised gas cylinder of ethane fitted with a two-stage pressure regulator and connected via 6mm PTFE tubing
- stainless steel bottle or glass bulb containing distilled de-ionised water

**Water preparation**

A water sample is prepared by filling a stainless steel or glass bulb with approx. 5 ml of distilled de-ionised water and connected to the gas line via a Swagelok fitting. The water must be freeze-pump-thawed three times to eliminate any dissolved gaseous impurities. To do this:

1. Using a small 1 L dewar, freeze the water in the bulb by gently immersing it in LN2. Ensure that gloves suitable for cryogenic use and goggles are worn and immerse very slowly to avoid excessive splashing as the LN2 boils on contact with the room temperature bulb.
2. Once completely frozen, pump away the volume of air above the frozen water via the gas line creating a vacuum in the bulb.
3. close the valves to the gas line and allow the water to thaw – releasing gases into the vacuum of the bulb/bottle. Avoid using a hot air gun or a hot water path to speed the thawing process if a glass bulb is being used.
4. Repeat steps 1 to 3 three times.

**Ethane preparation**

The PAC setup should include an in-line sorbent trap to remove flammable gases from the exhaust. This will also prevent ethane from becoming trapped in the oil of the rotary pump. Activated alumina should act as an effective molecular sieve to remove the small quantities of ethane being used. Note that the lower explosive flammability limit of ethane is 2.9% in air. With the small quantities of gas that are expected to pass through the system, even in the event of a complete leak from the vacuum line after the regulator, this limit will never be reached.

The following procedure should be followed to fill the gas line with ethane:

1. Vacuum leak test all the connections between the gas line and the ethane cylinder, including the regulator and thoroughly evacuate the line between the gas line and the cylinder.
2. Only when absolutely certain that there are no leaks, isolate the ethane line from the gas line with the on/off valve and fill the ethane line with 1 – 1.5 bar of ethane.
3. Isolate the gas line from the pump with the on/off valve and fill the gas line with <10 mbar ethane using a needle valve to control the flow, then close the needle valve and the on/off valve to the ethane line.
4. Continue with mixing with
5. Admitting each gas/vapour sample in turn to the desired mix of partial pressures within the mixing volume, keeping the total pressure below 10 mbar.

**Vapour/Gas dosing and mixing**

Gases are mixed in the gas line (with ethane:water ratios between 1:1 to 1:100) by first filling with a small quantity of ethane (few mbar), pumping out to the desired partial pressure if necessary, then 'topping up' with water vapour, by finely controlling the needle valve on the water line. The total pressure in the gas line is kept below 10 mbar. Further dilution is achieved by pumping out some of the mixture and 'topping-up' with more water until the desired ratio op partial pressures is achieved. If any ethane-rich mixes are required, reverse the order by filling the water first followed by ethane.

Once the gases have been prepared in the gas line, the mixing volume (consisting of a CF40 T-piece) is isolated prior to dosing. Dosing of gases is then carried out through a nozzle directed on to a pre-cooled ZnSe substrate within the UHV chamber, via the all-metal precision leak valve. Ice films are grown at a maximum rate of 1 µg second up to maximum thickness of ~1 µm. Total gas usage is typically less than 0.1 bar/L.

The ice films can be grown at various temperatures between 20 and 200 K and subsequently subjected to a heating ramp up to 250 K, by regulating the temperature of the substrate via a PID temperature controller. At the end of the experiment the substrate is cleaned by annealing to 300 K.

**ZnSe substrate**

An optically flat ZnSe substrate is attached to the cold finger inside the UHV chamber via a custom-built sample holder. Occasionally the substrate will need to be removed for cleaning purposes. ZnSe is toxic and gloves must be worn when handling. Substrates should be checked for chips or cracks when mounting or dismounting and any debris disposed of appropriately.

**Use of LN2**

Liquid nitrogen is used to freeze-pump-thaw water samples as well as for cooling the MCT detector of the FTIR spectrometer. Use a funnel to pour liquid nitrogen into FTIR slowly to avoid spillage onto the optics box and the surrounding equipment.

LN2 is decanted from a 25 L dewar situated in the lab into a small 1 L dewar. The 25 L dewar is filled from a pressurised vessel in a cage outside Robert Hooke building approximately once a month. For a detailed procedure on dispensing LN2 from a pressurised cylinder please refer to **RA860126**. The 25L dewar is mounted in a tipping trolley and care must be taken when decanting into a 1 L dewar to avoid splashing onto the floor or feet. Cryo-gloves and goggles must be worn at all times while handling liquid nitrogen in the lab. At no point should an open dewar of liquid nitrogen be left unattended. The lab is equipped with O2 alarms.

## Substance Information

**Product Name**
Ethane ( 00582 )

**Manufacturer**
Sigma-Aldrich Company Ltd

**Hazard Symbols**

**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 )

P410+403: Protect from sunlight. Store in a well ventilated place

| Application Method | Application Area | Frequency Of Use | Quantity Used | Duration Use |
|---|---|---|---|---|
| FTIR spectroscopy of pure, layered and mixed ethane and water ice films | Physical Sciences | 2-3 times per week | 0.1 mol/l | 4 months |

**Spillage, Storage & Waste**

**Spillage:** Spillage is unlikely as as vacuum lines and system will be carefully leak tested prior to flowing gas into an isolated part of the system, trapped at low (<10 mbar) pressured and released at a slow rate (1 µg/s) into a vacuum chamber. However, in the unlikely event of accidental release do not allow to accumulate where air or oxygen is present. Turn of valves immediately if leak is suspected. 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

**Accidental release measures**

6.1. Personal precautions protective equipment and emergency procedures: avoid breathing vapors mist or gas ensure adequate ventilation remove all sources of ignition evacuate personnel to safe areas beware of vapours accumulating to form explosive concentrations vapours can accumulate in low areas. 6.2 Environmental precautions: prevent further leakage or spillage if safe to do so do not let product enter drains 6.3 Methods and materials for containment and cleaning up: clean up promptly by sweeping or vacuum 6 4 reference to other sections for disposal see section 13.7

**Handling and storage**

7.1 Precautions for safe handling: avoid inhalation of vapour or mist keep away from sources of ignition, no smoking, take measures to prevent the build up of electrostatic charge. 7.2 Conditions for safe storage including any incompatibilities: store in cool place keep container tightly closed in a dry and well-ventilated place contents under pressure. 7.3 specific end uses no data available 8.

Always close all valves and keep sealed between uses. Secure gas cylinder in a stable position to prevent any slipping or falling

**Waste:** only limited waste ethane should be generated through the assessed procedure and this should be adsorbed onto an in-line trap before exhaust. The trap contents should be refreshed, with care taken to allow for emission of small quantities of ethane. Should usage prove to be greater than predicted (e.g. through some fault with the system), consider disposing of sorbent as chemical waste rather than refreshing as liberating large amounts of ethane may be unsafe. Any unused gas in the cylinder can be returned to the supplier

**WELS**

Contains no substances with occupational exposure limit values.

| Product Name | Manufacturer | Hazard Symbols |
|---|---|---|
| Distilled Water ( M1629-001 ) | In House | |

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| Application Method | Application Area | Frequency Of Use | Quantity Used | Duration Use |
|---|---|---|---|---|
| FTIR spectroscopy of pure. layered and mixed ethane and water ice films | Physical Sciences | less than once per month | 5 ml | 4 months |

**Spillage, Storage & Waste**

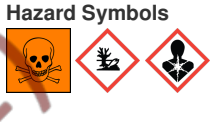
**Storage:** care should be taken to avoid contact with electronic equipment or incompatible substances. Water can be stored in any suitable container with a sealing lid or, less ideally, an open container where such contact is highly unlikely on spillage. Where a known grade of de-ionized water is required, it may advisable to take freshly purified water each time as effective purity will fall on prolonged air contact.

**Spillage:** spilled water should be mopped up with blue roll or similar (which can be disposed of in any appropriate bin). Do not leave large spills unattended. Where spills are near electronic equipment, switch this of first.

**Waste:** any waste water can be poured down the sink.

**WELS**

N/A

| Product Name | Manufacturer | Hazard Symbols |
|---|---|---|
| Zinc selenide ( 244619 ) | Goodfellow Cambridge Limited |  |

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| Application Method | Application Area | Frequency Of Use | Quantity Used | Duration Use |
|---|---|---|---|---|
| FTIR spectroscopy of pure. layered and mixed ethane and water ice films | Portable Astrochemistry Chamber, Perry D | less than once per month | single disk of 2 cm diameter x 2 mm thickness | 4 months |

**Spillage, Storage & Waste**

**Spillage:** ZnSe is a solid substrate. Spillage does not apply unless substrate is broken in a significant way. This is not expected to occur; seek advice if this does happen as fragments will likely be sharp and any dust formed will be toxic. Wear gloves when handling.

**Storage:** store in a cool, dry, place inside a tightly sealed container. ZnSe is moisture and air sensitive, so it is recommended to flush container through with nitrogen between uses. An appropriate desiccant may also be considered. Container must be labelled suitably for storage class TRGS 510: Non-combustible, acute toxic Cat. 1 and 2 / very toxic, hazardous materials

**Waste:** disposal of waste must be arranged through a license disposal company. Do not throw into general lab waste. Keep any collected waste in a tightly sealed and labelled container.

**WELS**

0.1 mg/m [UK. EH40 WEL - Workplace Exposure Limits]

## Task Related Hazards & Controls

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

Low 10 High

REQUIRED CONTROLS

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

Low 4 High

**Hazard Description**

Ethane

H220: Extremely flammable gas

**Controls In Place**


Secure gas cylinders


P403 Store in a well-ventilated place


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


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


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


Inspection: Check the sorebent foreline trap on the rotary pump and change contents as necessary.


Evacuate all gas lines and regulators when not in use, after ensuring that the valve to the cylinder is securely closed.


Do not open the gas cylinder and allow any gas flow until all vacuum lines, connections, valves and the regulator are thoroughly leak-tested.


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


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.

**Emergency Controls In Place**


P377 Leaking gas fire: Do not extinguish, unless leak can be stopped safely.


In case of an emergency involving accidental and uncontrollable leaks that cannot be contained, evacuate the area immediately.


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

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

Low 10 High

REQUIRED CONTROLS

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

Low 5 High

**Hazard Description**

Ethane cylinder:
H280: 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.

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: **Medium Risk** (8/25)

Low 8 High

REQUIRED CONTROLS

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

Low 3 High

**Hazard Description**

zinc selenide
Warning; Toxic material

**Controls In Place**

Ensure that zinc selenide substrates are stored in a secure location, in a clearly labelled container

Inspection: Check substrate for any cracks or chips when mounting or dismounting from the sample holder

Wear protective gloves when changing the substrate. Do not use the same gloves to handle any other substances

**Emergency Controls In Place**

Wear a mask if any breakage, shattering of zinc selenide has occurred - avoid inhaling any resultant dust during cleaning.

Wash your hands or any area of skin thoroughly with soap and water that has come into contact with zinc selenide

If any ZnSe is thought to have been ingested, contact a medical professional and poison centre immediately

Before Controls: **Medium Risk** (8/25)

Low 8 High

REQUIRED CONTROLS

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

Low 3 High

**Hazard Description**

Danger of cumulative effects (selenides)

**Controls In Place**

Information regarding repeat exposure effects is given on the material data sheets and in the "Health Surveillance" section of this assessment. Ensure that operators are familiar with potential symptoms and descriptions before starting work.

**Emergency Controls In Place**

Do not ignore any potential symptoms of cumulative exposure effects. Contact occupation health and a medical professional if experienced.

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

Low 2 High

REQUIRED CONTROLS

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

Low 1 High

**Hazard Description**

zinc selenide
Waste/Spillage substance material chemical environmental environment

**Controls In Place**

Follow disposal procedures listed for ZnSe. Do not dispose of with general lab waste

## General Controls & Emergency Controls

**Controls In Place**

Wear safety glasses when handling pressurised gases or dangerous compounds.

Wear lone worker alarm when necessary

Secure gas cylinders

Wear protective gloves when handling of zinc selenide. Nitrile gloves should be considered sufficient to give full protection. Dispose of gloves in lab bin after use

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

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

**Emergency Controls In Place**

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

Sound Alarm - if O2 alarm sounds evacuate

If in doubt regarding exposure to any toxic and/or harmful compounds, seek medical advice.

Evacuate to assembly point should any alarm trigger

Sound the alarm in case of any accidental or uncontrollable leak develops, but only after it is safe to do so.

Seek advice/training when in doubt about any part of the vacuum or cryogenic equipment from trained individuals.

## Health Surveillance

Zinc selenide can cause damage to organs through repeat exposure. Symptoms, as described in material safety data sheet, are:

"Acute selenium poisoning produces central nervous system effects , which include nervousness, convulsions, and drowsiness. Other signs of intoxication can include skin eruptions , lassitude, gastrointestinal distress, teeth that are discolored or decayed, odorous ("garlic") breath, and partial loss of hair and nails . Chronic exposure by inhalation can produce symptoms that include pallor, coating of the tongue, anemia, irritation of the mucosa, lumbar pain, liver and spleen damage, as well as any of the other previously mentioned symptoms . Chronic contact with selenium compounds may cause garlic odor of breath and sweat, dermatitis , and moderate emotional instability."

Should any sign of these symptoms arise contact occupational health immediately and ensure they are aware that medical attention is required. Cease all work with acids or selenium bearing compounds immediately until a medical professional has confirmed this will be safe to resume following a review of procedures.

## Action Management

| Ref | Action Type | Priority | Target Date | Action Owner |
|---|---|---|---|---|
| 439648 | Review and Publish | High | 2020-09-07 10:40:28 | Anita Dawes |
| **Action Title** | Check risk assessment | | | |
| **Description** | Check the risk assessment before publishing and approvals | | | |
| **Status** | Incomplete | | | |

| Actionees | Email |
|---|---|
| Thomas Webley | thomas.webley@open.ac.uk |
| Lily Hills | lily.hills@open.ac.uk |
| Vincent Deguin | vincent.deguin@open.ac.uk |

## Linked Documents

| Reference | Title | Uploaded By | Uploaded At |
|---|---|---|---|
| 67244 | RA83550 Use of portable vacuum chambers in astrochemistry laboratories | Anita Dawes | 2020-09-03 10:36:54 |
| 67245 | Dispensing liquid nitrogen and transfer of liquid nitrogen from self pressurising dewar to other containers | Anita Dawes | 2020-09-03 10:37:52 |