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[Home](#) > Chemistry Experiment using reflux and distillation

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## Chemistry Experiment using reflux and distillation

Posted by Anonymous on Tue, 2016-06-21 16:09

Chemistry Experiment using reflux and distillation: Is there an organic practical that uses two of reflux, distillation and liquid-liquid extraction that does not require a fume hood? The most common practical is esterification, but as I understand it that needs a fume hood and probably a water bath, and at present I don't have access to either. Or can I do it without those since it is under reflux?

**Voting:**



No votes yet

**Australian Curriculum:**

Chemical Sciences

**Year Level:**

Senior Secondary

**Laboratory Technicians:**

Laboratory Technicians

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Showing 1-1 of 1 Responses

## Chemistry Experiment

Submitted by sat on 22 July 2016

Answer reviewed 8 March 2023

The absence of access to a fume cupboard is very challenging for a secondary science facility because this is required for many chemical procedures. It is recommended that activities involving reflux, distillation and solvent extraction of organic substances **should be conducted under an operating fume cupboard** due to the hazardous nature of the chemical substances.

### Making esters

The preparation of esters can be conducted safely on a microscale level in a well-ventilated room without involving reflux and distillation processes. The Royal Society of Chemistry's<sup>1</sup> procedure using a microscale method is safe with respect to the conditions required and volumes of reagents involved, and enables students to characterise their product esters by odour.

### Water baths

When working with flammable liquids, it is essential that there be no naked flames from Bunsen burners. For heating reactions up to 100°C, water baths can be improvised using a suitable container, such as a glass beaker or metal baking pan, placed upon a hotplate. For efficiency, the water can be heated in a kettle before filling the bath, and the heat maintained with the hotplate.

### Reflux

Access to a fume cupboard is recommended for the dispensing of the chemicals and for the refluxing procedure. However, when carried out correctly, the refluxing procedure should release minimal vapours into the lab. Care should be taken to ensure that water is flowing through the condenser; a slow continuous flow of water is sufficient. If the mixture is overheated, vapours may escape from the top of the condenser, in which case, the heating temperature should be reduced. Vapours can also escape if the flask and condenser are not fitted together correctly. A small amount of vacuum grease (silicone grease) applied to the ground glass joint will also help to prevent the escape of vapours from this joint.

### Safer alternative activities

It is challenging to find suitable and safer alternatives that do not require the use of a fume cupboard. We have previously answered similar or related questions that may be helpful, as follows:

- [Reflux and distillation](#) for recommended methods for heating.
- [Esters - preparation, properties and disposal](#) and [Organic chemistry](#) for general guidance and proper disposal methods.

### Additional information

#### Chemicals used and produced in esterification

- Concentrated sulfuric acid is extremely corrosive to skin and eyes and should be handled with great care. Measures should be taken to prevent inhalation of the fumes from the concentrated acid. PPE should be worn and the acid handled in an operating fume cupboard or where there is very good ventilation.
- The alcohols that are mostly used for esterification reactions in schools are methanol, ethanol, propan-1-ol, butan-1-ol, pentan-1-ol, octan-1-ol and iso-amyl alcohol. These alcohols are flammable or combustible and should be kept away from all naked flames and sources of ignition. The alcohol vapours can cause dizziness and drowsiness. Methanol is highly toxic if swallowed or inhaled. They can all cause irritation and most can cause serious eye damage. They have all been categorised as toxic, to varying degrees, and accordingly, should all be handled with care. Ensure that all people handling them observe good laboratory hygiene and wash their hands before leaving the laboratory.

- Commonly used carboxylic acids are glacial acetic acid and propionic acid. They are highly corrosive to skin and eyes, and toxic by inhalation.
- The esters formed are flammable or combustible. In general, esters can cause irritation to the eyes and respiratory tract, and may cause drowsiness or dizziness.

## References

<sup>1</sup> Royal Society of Chemistry, 2023, *Experiments: Making esters from alcohols and acids*, RSC Education website. <https://edu.rsc.org/lcredir/learn-chemistry/resource/res00001743/making-esters-from-alcohols-and-acids> (Accessed March 2023)

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