Supporting Technical Education Teaching:

**Curriculum Resources**

Teaching Guide

Topic: Refluxing

Version information

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| **Route** | Health & Science |
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| **Topic** | Refluxing |
| **Specification coverage** | **Performance outcome 1:** Perform a range of appropriate scientific techniques to collect experimental data in a laboratory setting, complying with regulations and requirements  **Core scientific knowledge** K1.20, K1.21, K1.22, S1.73, S1.79, S1.84, S1.85  **A10 Experimental equipment and techniques** A10.3 |

This resource is part of a series of materials to support technical education teaching. The approach to developing the materials draws from research led by Professor Kevin Orr that sets out a model for understanding of technical education pedagogy.

The curriculum development begins with the knowledge that students are working to learn and apply. Teachers draw from their subject and industry expertise, and their knowledge of their students, to make decisions about the core concepts the curriculum will focus on, how they will sequence these concepts, and the activities that are selected to support students’ learning. The decisions behind the resources suggested in this topic are the result of choices made by the curriculum development team, which will be reviewed and improved by teachers’ decision-making and ongoing reflection in their own circumstances.

The materials also seek to support teachers in bringing classroom and industry closer together, by providing assets that draw from authentic industry materials, and using opportunities to capture workplace practice that can be shared with students.

Materials for other topics are available at: [www.technicaleducationnetworks.org.uk](http://www.technicaleducationnetworks.org.uk)

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HEALTH AND SAFETY

This topic has been safety checked but not trialled by CLEAPSS.

It is assumed that activities outlined in this Teaching Guide will be undertaken in suitable facilities or work areas and that good practices, appropriate use policies and procedures will be observed. Teachers should consult their employers’ risk assessments before use and consider whether any modification is necessary for the particular circumstances of their own class/institution.

For practical activities, the Technical Education Networks programme has tried to ensure that experiments are healthy and safe to use in colleges and schools, and that any recognised hazards have been indicated together with appropriate control measures (safety precautions). It is assumed that experiments and activities will be undertaken in suitable laboratories or work areas and that good laboratory practices will be observed. To access the CLEAPSS materials in this suite, institutions will need to be a member of CLEAPSS. Further details are available at www.cleapss.org.uk If necessary, CLEAPSS members can obtain further advice by contacting the Helpline by email at science@cleapss.org.uk or on 01895 251496.

Acknowledgements

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**Introduction**

This document for teachers outlines both the topic area covered, and approach to using the suite of resources and assets for each lesson. Unless otherwise stated, definitions of key terms have been developed by the authoring team and reviewed in the context of the activities. Teachers may choose to revise definitions as necessary.

# Topic purpose

This topic is a focus on the practical technique of refluxing. It introduces the concept and looks briefly at the theory and ensures students develop practical skills to describe, perform and evaluate the refluxing technique. There are opportunities for students to experience refluxing in the context of industry and to discuss its importance.

There are two lessons, which are assumed to be 1.5 hours each and designed to be taught one after the other. Teachers may want to adapt the suggested sequencing of concepts and activities as appropriate for students and circumstances. The lessons are broken down to provide teacher flexibility on the depth covered in the activities; lessons can also be split over multiple shorter lessons if required. There are extension and support suggestions and alternatives to practical activities depending on the provider setup.

There are also opportunities to build several essential skills that are developed during the course and General competencies for maths, English and digital.

The content in the lessons can be reinforced throughout the course to support students’ learning. For example, refluxing appears in numerous places in the specification, and the process of performing a reflux employs many practical skills such as the physical manipulation of jointed glassware. Therefore, this topic can be used to strengthen students’ practical skills as well as develop theoretical skills in the core content. The theoretical understanding of changes of state, boiling points, evaporation-condensation and others are core concepts that are relied upon for this lesson. Understanding a reflux leads naturally into understanding distillation. When discussing a forthcoming industry placement, students can investigate the practical skills that may be required of them and note this learning in their logbook. For example: [support.tlevels.gov.uk/hc/en-gb/articles/360015345420-Industry-placement-logbook-for-students](https://support.tlevels.gov.uk/hc/en-gb/articles/360015345420-Industry-placement-logbook-for-students)

# Industry importance

Refluxing is an important scientific technique that is key in many different industrial processes.

There are many benefits to using a reflux in both batch and continuous processes, including: the ability to specify and maintain the high reaction temperatures by choice of solvent; the added mixing that occurs naturally during the process; the maintaining of consistent reaction concentrations improving the reliability of batch processes; decreased financial and environmental raw material costs; and the ability to leave the reaction for long periods due to minimal solvent/reactant loss. All of this allows for reactions with high activation energies to be run with desirable and reliable rates of reaction, with minimal raw material cost, and increases the efficiency of batch and continuous processes.

Refluxes also improve the efficiency of distillation processes in the petrochemical, beverage and natural gas processing industries, increasing product purity and enhancing separation, whilst also decreasing processing costs. This is achieved because the reflux process – the act of heating a substance to above its boiling point, allowing it to rise, cool and then drop down the column to be heated again – cools down vapours as they rise through the column. This increases the efficiency of the separation within the distillation tower, allowing for a simpler distillation setup.

Many of the fundamental scientific principles, practical skills and safety considerations that must be understood for a reflux are relied upon when discussing the use of reflux and distillations in other practical experiments, and more advanced topics in higher education settings, such as Soxhlet extraction and use of a Dean-Stark apparatus.

*“Refluxes are a key practical skill for any chemist to master because they rely on your ability to manipulate industry standard equipment, troubleshoot problems within practical procedures, understand safety considerations of handling heated chemical systems and apply a range of theoretical concepts to your work. By understanding reflux, you open the doors to a range of other techniques and chemical manipulations which all operate at elevated temperatures across a range of chemistry sub-disciplines.”*

***Dr Alex Wright, Lecturer in Chemistry, University of Kent***

# Industry links

**General information on refluxing and setups**

* General information from ChemEurope and valuably detailed theory on refluxing in industry, and how and where it is used: www.chemeurope.com/en/encyclopedia/Reflux.html
* University of York’s instructional video on how to set up and take down reflux glassware for a generic reflux: [youtube.com/watch?v=mrD6dP3TLNw](https://www.youtube.com/watch?v=mrD6dP3TLNw)
* CapilanoUChemlab’s instructional video on how to set up and take down a generic reflux in the laboratory, including some safety considerations:

[www.youtube.com/watch?v=\_SzYqPdUkFQ](http://www.youtube.com/watch?v=_SzYqPdUkFQ)

* ChemSurvival’s instructional video that explains why boiling a solution for long periods of time can be problematic, and how a generic reflux setup can be used to overcome these issues: [www.youtube.com/watch?v=6VbNyFuYQMU](http://www.youtube.com/watch?v=6VbNyFuYQMU)
* An overview of the reflux practical setup written by Lisa Nichols from Butte College, with detailed step-by-step how-to instructions, including some common setup errors:

<https://chem.libretexts.org/Bookshelves/Organic_Chemistry/Organic_Chemistry_Lab_Techniques_(Nichols)/01%3A_General_Techniques/1.04%3A_Heating_and_Cooling_Methods/1.4K%3A_Reflux>

* Some examples of reflux mistakes, the consequences of them, and how to overcome them, written by Radleys: [www.radleys.com/blog/reflux-reaction-mistakes-to-avoid/](http://www.radleys.com/blog/reflux-reaction-mistakes-to-avoid/)
* An animation to support learners who aren’t confident with the particle theory during changing states: <https://www.youtube.com/watch?v=hkBrw2fG75U>
* An interactive lab primer, created by the Royal Society of Chemistry, that gives core safety information, skills and apparatus to support as a visual aid to common laboratory techniques: [edu.rsc.org/resources/interactive-lab-primer/1064.article](https://edu.rsc.org/resources/interactive-lab-primer/1064.article)

**Reflux practicals**

* An educational and instructive video from Malmesbury Education showing how to perform a reflux practical oxidising ethanol to produce ethanoic acid with some reflux and oxidation theory too: [www.youtube.com/watch?v=aUEpAEaSsBU](http://www.youtube.com/watch?v=aUEpAEaSsBU)
* An interactive ‘screen experiment’ by the Royal Society of Chemistry, which allows students to ‘perform’ a practical to synthesise aspirin by completing an online activity:

[edu.rsc.org/resources/aspirin-screen-experiment/1644.article](https://edu.rsc.org/resources/aspirin-screen-experiment/1644.article)

* A Royal Society of Chemistry video resource which shows the difference between reflux and distillation and includes instructions for completing both processes in video form:

[edu.rsc.org/practical/reflux-and-distillation-practical-videos-16-18-students/4012294.article](https://edu.rsc.org/practical/reflux-and-distillation-practical-videos-16-18-students/4012294.article)

* An article from the Royal Society of Chemistry which includes instructions for a reflux as part of the production of 2-hydroxybenzoic acid, used to produce aspirin:

[edu.rsc.org/resources/the-preparation-of-2-hydroxybenzoic-acid-16-18-years/4017310.article](https://edu.rsc.org/resources/the-preparation-of-2-hydroxybenzoic-acid-16-18-years/4017310.article)

**Microscale**

* An educative and instructional video from Bob Worley from CLEAPSS with a method and minor explanations about how to perform a microscale reflux reaction to prepare esters of butanol: [www.youtube.com/watch?v=YKAgmQZHUkc](http://www.youtube.com/watch?v=YKAgmQZHUkc)
* Information and links to support the process of using microscale reflux chemistry to make aspirin, written as a LinkedIn post by Bob Worley from CLEAPSS, including a link to the Royal Society of Chemistry resource for the microscale reaction to produce esters: [www.linkedin.com/posts/bob-worley-12034631\_cleapss-activity-7157307723355684864-hHaQ/](http://www.linkedin.com/posts/bob-worley-12034631_cleapss-activity-7157307723355684864-hHaQ/)

**Other**

* Materials for topics to support Health and Science T Levels are also available on the NCFE YouTube Channel available at: <https://www.youtube.com/playlist?list=PL05CIlRfHw9iJ-Ga_OSWeRXU_ZDyaKJ5v>

# Prior learning

Students will likely have met the theoretical content behind the process of reflux at GCSE. However, it is always worth examining the depth of existing knowledge before this topic, as students will find the use of reflux much more accessible if they are familiar with the particle model and the theory of changing states. An understanding of the transfer of energy, and the names of common glassware will also be needed.

These lessons do not cover distillation in any detail, but lots of reflux practicals involve it and the two processes are very closely linked in industry. Distillation is also met more often in GCSE so students may already be more familiar with it. An understanding of distillation will help support students to understand how it differs from reflux – reflux is used for preparations/reactions, whereas distillation is commonly used for purifications/separations.

# Accessibility

The teaching materials have been designed to provide teachers with a flexible framework, including different approaches to activities, suggested consolidation activities to further embed knowledge and adaptable study questions to assess learning. As with all resources, teachers will wish to consider the specific needs of their students when using the materials, including Special Educational Needs and Disabilities (SEND). Although content has been reviewed, accessibility in externally linked resources cannot be guaranteed.

These resources are designed to be taught in a classroom or laboratory setting, with students able to work on their own to complete practicals, or in pairs/groups to complete discussions and paired/grouped tasks. If this is not possible, the paired tasks can be completed as solo research tasks instead, to allow the student the opportunity to engage with a variety of ideas and expose them to as large a variety of industries and environments as possible. Practicals can be performed solo, in pairs, or in groups, and the equipment can be adjusted to account for availability.

Students' base level of science knowledge may affect how much explanation is needed to make appropriate industry links. Drawing on the students' own experiences, including from their industry placements, will support this. Students' understanding and experience of the world of work will be varied, which may mean they find applying the content to an industry environment challenging. Engaging in whole-group discussions where appropriate and sharing personal and students' experiences will support learning.

**Learning outcomes and specification coverage**

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| **Lesson** | **Learning outcomes** | **Specification coverage** | **Skills and general competencies** | **Links to other specification content** |
| **1** | Students will be able to:   * Write word and symbol equations to show the oxidation of alcohols to form carboxylic acids * Describe the uses of refluxing during organic synthesis * Identify and justify the use of a reflux reaction * Identify the appropriate equipment and explain the correct setup for a reflux reaction | **K1.20** The word and symbol equations to show reactions of the following organic compounds:   * alcohols (methanol, ethanol, propanol and butanol): oxidation to a ketone or carboxylic acid with the use of [O] as the oxidising agent   **K1.21** The possible uses of the following techniques used during organic synthesis:   * refluxing   **K1.22** The oxidation and reduction process  **A10.3** Applications of the following equipment when undertaking scientific techniques:   * glassware * scientific balances | Skills  **S1.73** Apply scientific knowledge when undertaking scientific techniques  **S1.79** Use the following practical scientific techniques to prepare, isolate and separate materials:   * + refluxing   **S1.84** Select appropriate equipment to complete practical scientific techniques:  General competencies  English:  **GEC2** Present information and ideas  **GEC4** Summarise information/ideas  Maths:  **GMC1** Measuring with precision  **GMC10** Optimising work processes | **K1.2** How to use resources efficiently when performing scientific techniques  **K1.54** The purpose of laboratory techniques used in the science manufacturing environment  **K1.59** A range of laboratory equipment that is used to analyse BOD, COD and TOC  Occupational specialism: technical metrology:  **K1.20:** How to apply best practice principles in measurement |
| **2** | Students will be able to:   * Explain the use and application of glassware, thermometers, and balances when undertaking scientific techniques * Identify the appropriate equipment and explain the correct setup for a reflux reaction * Demonstrate practical technical competence in the use of equipment when performing a reflux * Safely perform a reflux using appropriate personal protective equipment (PPE) | **K1.21** The possible uses of the following techniques used during organic synthesis:   * refluxing   **A10.3** Applications of the following equipment when undertaking scientific techniques:   * glassware * scientific balances samples | Skills  **S1.73** Apply scientific knowledge when undertaking scientific techniques  **S1.79** Use the following practical scientific techniques to prepare, isolate and separate materials:   * refluxing   **S1.84** Select appropriate equipment to complete practical scientific techniques  **S1.85** Demonstrate practical technical competence in the use of equipment  General competencies  English:  **GEC2** Present information and ideas  **GEC4** Summarise information/ideas  Maths:  **GMC1** Measuring with precision  **GMC10** Optimising work processes | **K1.1** How health, safety and environmental practices are applied when performing scientific techniques  **K1.2** How to use resources efficiently when performing scientific techniques  **K1.54** The purpose of laboratory techniques used in the science manufacturing environment  **S1.68** Work safely in a laboratory when performing specific scientific techniques  **S1.69** Comply with relevant health and safety legislation and regulations  **K1.59** A range of laboratory equipment that is used to analyse BOD, COD and TOC  Occupational specialism: technical metrology sciences:  **K1.20** How to apply best practice principles in measurement |

**Lesson guidance**

# Lesson 1: Reflux and oxidation

This lesson introduces and delves into the process of reflux and oxidation. It allows students to experience what a reflux practical looks like, to discuss the theory behind a reflux and to explore why reflux is used in industry. Students will also be asked to evaluate refluxing setups and to discuss and justify the use of reflux. The oxidation of alcohols is also covered, specifically within the context of refluxing to produce a carboxylic acid, and the word and symbol equations for this reaction are discussed.

## Preparation

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| **Resources provided** | * L1 Slide deck * L1 Worksheet – for all activities * L1 Worksheet answers * L1 Teacher support sheet |
| **Equipment needed** | For the demonstration as given:   * Liebig condenser * Round-bottom flask and heating mantle OR pear-shaped flask and water bath * Ice water bath * Anti-bumping granules * Clamp and stand |
| **Safety factors** | Ensure risk assessments have been conducted and checked by an appropriate practitioner.  Acidified potassium dichromate is an oxidising agent. It is an irritant and health hazard. Wear gloves whilst handling and avoid skin contact.  Reflux reactions involve heating glassware and reactants to high temperatures and can pose risk of burns if not monitored and handled correctly. Reflux apparatus should be left to cool before handling.  Whilst watching the demonstration, students should stand well back and wear safety goggles in case of bumping/spillage.  Safety goggles should be worn at all times whilst performing practicals and demonstrations.  Ethanol is flammable so should be kept away from naked flames. It burns with an invisible flame. |
| **CLEAPSS references** | Acidified potassium dichromate is an oxidiser, toxic and a health hazard, HC078c  Ethanol is flammable, an irritant and toxic, HC040A  Ethanoic acid > 1.7 M is an irritant, HC038a |
| **Prior learning** | * As well as the prior learning on page 8, students should be familiar with the basic structures of straight-chain alcohols and carboxylic acids (up to butanol/butanoic acid, the 4th member of the alcohol/carboxylic homogenous series), and understand the use of structural formulae and how to construct a word equation. * An understanding of particle theory and state changes, and how the movement, arrangement, and separation of particles in the three states of matter change during state changes is needed to grasp the theory behind reflux. * Students should be familiar with the concept of a reversible reaction, and the effect of temperature on some reversible reactions, as this concept is covered but not explained in any detail in this lesson. * Students can draw on previous experience of using glassware/apparatus and performing practicals to inform answers to questions during the teacher demonstration and to help them apply the concept of reflux to industry situations. |
| **Common misconceptions** | * Students often struggle to understand the difference between distillation and reflux, and the purpose of reflux. There is often confusion as to why refluxing is needed and how it differs to a simple distillation and product collection. Emphasise that, in general, distillations are to separate and refluxes are to react. * The theory of state change between liquid-gas is a confusing concept for students as it is abstract, and they sometimes forget that boiling point and condensing point are the same thing. * The particle model for changing states is full of misconceptions. Often students consider a ‘solid/liquid/gas’ as a type of substance rather than a state of matter. |
| **Accessibility** | * Seek to ensure wide representation for any visiting speakers and case studies used (where appropriate). * Ensure groups are diverse and balanced and consider pairing students with peer buddies if needed. * Teachers may wish to use pair work to help students of different abilities to support one another. * Ensure students have access to IT equipment with a range of software available (or links to appropriate websites). |

## Activity guide

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| **Introduction**  SUGGESTED TIME:  20 minutes  RESOURCES:   * L1 Slide deck – Slides 2–5 * L1 Worksheet | * Go through the learning outcomes for this lesson with students on Slide 2. * Ask students to THINK, PAIR, SHARE (think on their own, then share the idea with a partner, then ask students to share their ideas as a whole class) about why heat is required in some chemical reactions (Slide 3). Discussion points could include:   + Some chemical reactions/processes have high activation energies or require large transfers of energy. Energy in the form of heat can be used to overcome these high activation energies, or to help provide the energy for the process.   + In reversible reactions, in which the forwards reaction is endothermic, increasing the temperature will push the equilibrium towards the products (the thermodynamic product in that case), so higher product yields can be achieved in shorter times.   + Increasing the temperature will also increase the kinetic energy of the reactant particles, which will increase the rate of any chemical reactions occurring. This could mean a larger yield in a shorter period. * Show students the scenario on Slide 4 and ask them to THINK, PAIR, SHARE what issues they can foresee about heating reactions for long periods of time. If students are struggling, introduce the real-life scenario of a pot of pasta left on the hob on a high heat for a long time and ask what issues they can foresee. Highlight that eventually the reaction vessel would boil dry as all the ethanol (or water in the pasta pot example) boils off/evaporates – this will stop the reaction and could be a safety concern. Suggest that a method for overcoming this issue could include returning the evaporated solvent to the reaction vessel, i.e. a reflux. * Give a brief explanation of what reflux is and when it is used: reflux is used to heat up and hold reactions with volatile solvents/reactants at high temperatures for long periods of time, without loss of solvent/reactants. * Show students a YouTube video (Slide 5) ([www.youtube.com/watch?v=\_SzYqPdUkFQ](http://www.youtube.com/watch?v=_SzYqPdUkFQ) 5.5 mins) of a traditional reflux setup and ask them to fill in the labels on the diagram on the Worksheet Introduction activity as they watch the video. The video can be paused periodically to allow students the time to identify and label the equipment. * Check the students’ understanding of: ‘bumping’, solvents and their use, volatility and boiling/evaporation. Check students have correctly labelled their setup diagram. * Students may be familiar with the Liebig condenser from GCSE, where it is used in distillation. Briefly mention the difference between reflux and distillation – reflux is a potentially infinitely long **reaction** with a continuous boiling and condensing of the solvent/volatile substances, whereas distillation involves **separating** off substances once they have reached their boiling point/evaporated. |
| **Activity 1 – Reflux demonstration**  Suggested time:  25 minutes  Resources:   * L1 Slide deck – Slides 6–10 * L1 Worksheet * L1 Teacher support sheet * Equipment outlined in L1 Activity 1 Teacher support sheet | * Follow the demonstration given on the Teacher support sheet to oxidise ethanol to ethanoic acid (Slide 6). Teachers might like to watch the following YouTube video in advance to prepare for the demo: [www.youtube.com/watch?v=aUEpAEaSsBU](http://www.youtube.com/watch?v=aUEpAEaSsBU). This can also be played to the class if there is insufficient time/equipment/space to perform a demo. * Whilst performing the demo, discuss the theory behind reflux and how it works, including why there is minimal reactant/product loss: * The reaction mixture heats up, a reactant/solvent will then reach its boiling point, then it boils/evaporates and rises up the condenser. The condenser is cooling the air/gases inside its inside sleeve, so it cools down the vapours to below the boiling point. They condense, and droplets fall back down into the reaction mixture. If the equipment is set up correctly, there is very little opportunity for vapours to escape. * If students are not confident with the particle theory during changing states, talk through particle theory using the animations in this video: <https://www.youtube.com/watch?v=hkBrw2fG75U> * Equipment points to discuss: The use of a heating mantle rather than Bunsen burners – Bunsen burners are not very efficient at heating (lots of heat loss to the environment and they are an inconsistent source of heat). They are also very unsafe if using flammable solvents so are not usually used in industry – heating mantels/hotplates, which are safer, more efficient and a more consistent and uniform source of heat, are used. * Discuss the different ways the Liebig condenser is used in reflux and distillation.  Reflux: the condenser is set up vertically above reaction vessel to allow cooled vapours to drop back down into the flask – very little loss of vapour. Distillation: the condenser is set at an angle to allow the separation of the mixture – the substances in the mixture boil at different temperatures, form a gas and condense in the condenser to form a liquid, which is collected. * Talk through the reaction: Acidified potassium dichromate (VI) as an oxidising agent, which oxidises the ethanol (an alcohol) to form ethanoic acid (a carboxylic acid). The reaction needs prolonged heating, so reflux is used to minimise the loss of the reactants/product. * Students then complete tasks 1 and 2 on the Worksheet, which are shown along with the answers on Slides 7–10. * In this reaction, the ethanol is gaining oxygen to form a carboxylic acid (it goes from one C-O single bond in the compound, to one C=O double bond and one C-O single bond, so increases the bonds to oxygen, losing hydrogen and gaining oxygen). Point out this structural change on the diagram on Slide 9. The hydrogen atoms lost in the oxidation of ethanol are also oxidised forming water, H2O. * NOTE: if reflux is NOT used (and only distillation used), the ethanol might not oxidise all the way to a carboxylic acid and will likely only oxidise to an aldehyde. * Students could be shown the video to help support them to make improvements to their explanations in Task 1 and Task 2. It shows a version of the reflux setup and explains the theory of reflux, including information on and animations of the state changes that occur. Show this video from 1:10 to the end: <https://www.youtube.com/watch?v=b6xFAEkjmGg>. |
| **Activity 2 – Reflux in industry**  Suggested time:  15 minutes  Resources:   * L1 Slide deck – Slides 11–12 * L1 Worksheet | * As a class, watch the short video on reflux in industry (Slide 11 <https://vimeo.com/1060452181>) and ask students to use it to complete Activity 2 on the Worksheet (identifying three different uses of reflux in industry and undertaking some research to explain how the use of reflux is beneficial in each case). Reveal some suggested answers on Slide 12. * Discuss that reflux in industry is useful because the refluxing mechanism allows the reaction mixture to be fairly continuously mixed, encouraging a higher product yield and faster rate of reaction, keeping costs lower and maximising potential profits. * Other points for discussion on why reflux is useful could include:   + Allows potentially harmful solvents to be heated but kept out of the environment.   + Stops the concentrations of the components from being changed significantly when heated due to loss of solvents or reactants from evaporation, which keeps costs of raw materials low, yield of product high due to lack of loss of reactants, and increases sustainability by minimising solvent usage.   + Allows reactions with high activation energies to be kept safely at high temperatures for long periods of time, without worry of boiling dry, to keep the rate of the reaction high.   + Allows exothermic reactions to stay safely quenched or at an easily maintained temperature due to the continuous boiling and condensing of liquids/gases. * The main point to get across is that refluxing is used for ‘long reactions with volatile components’. Show that reflux is used in industry for both very small, and extremely large-scale reactions. For example, reflux is a core technique used in the synthesis of specialist novel drug molecules in the pharmaceutical industry such as Olaparib (anti-cancer); it supports and improves the efficiency of the separation processes in alcohol distillation; and then also in the separation of substances with more widespread and high-throughput products (produced or processed quickly and efficiently), such as renewable plastics. |
| **Activity 3 – Troubleshooting reflux**  Suggested time:  15 minutes  Resources:   * L1 Slide deck – Slides 13–14 * L1 Worksheet * L1 Worksheet answers | * Students complete Activity 3 on the Worksheet, which are troubleshooting examples to solve. They also lend well to small group discussion. Check student answers using the Worksheet answers. * If time allows, as a whole class, examine the five scenarios (A–E) on the interactive exercise linked on Slide 14, and here: [www.chemtube3d.com/reflux-apparatus-360-interactive-exercise/](http://www.chemtube3d.com/reflux-apparatus-360-interactive-exercise/) * Ask students to decide in pairs which is the correct setup and use class discussion to identify and correct the other four setups. The correct setup is setup A. There are hints on the website to help students if they need them. |
| **Plenary**  Suggested time:  15 minutes  Resources:   * L1 Slide deck – Slides 15–16 * L1 Worksheet * L1 Worksheet answers | * Ask students to answer the eight plenary questions from the Worksheet. The correct answers can be found in the answer sheet. * These questions should give insight into any gaps in knowledge or understanding from this lesson. * Revisit the learning objectives on Slide 16 to summarise the lesson before introducing the independent consolidation activity. |
| **Follow-up / consolidation** (to be completed outside of lesson)  Suggested time:  20 minutes  Resources:   * L1 Slide deck – Slide 17 | * Ask students to research the use of a waterless air condenser – a ‘greener alternative’ to using a (Liebig) water condenser in a batch process laboratory. * They should write a short article about this alternative to go in a company bulletin. * The article should evaluate and justify its use in a laboratory setting. |

# Lesson 2: Refluxing practical

This lesson allows students to develop the practical skills involved in the process of reflux. They experience what a reflux practical looks like and, where appropriate, this gives them the opportunity to follow a method to complete one. Students will also be asked to evaluate refluxing setups and possible errors.

## Preparation

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| **Resources provided** | * L2 Slide deck * L2 Teacher support sheet * L2 Worksheet |
| **Equipment needed** | For the reflux setup with errors:   * Round-bottom flask/pear-shaped flask * Conical flask * Heating mantle/hotplate stirrer * Anti-bumping granules or magnetic stirrer bar * Clamps, stand, and bosses * (Liebig) condenser and tubing * Quickfit thermometer stopper and thermometer * Lab jack   Reflux practical option 1:   * 50 cm3 round-bottom flask * Anti-bumping granules (if not using a magnetic stirrer bar) * Vegetable oil * Heating mantle/hotplate stirrer (and stirrer bar) * Lab jack * (Liebig) condenser and connecting tubes * 100-1000 µL autopipette (or 10 cm3 syringe if autopipette unavailable) * Methanol * 4 mol dm-3 sodium hydroxide solution * Boiling tube * Clamp, stand, and bosses * Access to a 2 d.p. balance and cork ring * Sample vial * Optional: glass wool, plastic pipette, heat proof mat, matches   Reflux practical option 2:   * 100 cm3 round-bottom flask * Anti-bumping granules (if not using a magnetic stirrer bar) * Heating mantle/hotplate stirrer * 5 cm3 graduated cylinder * Ethyl benzoate * Lab jack * (Liebig) condenser * 50 cm3 graduated cylinder * 2 mol dm-3 sodium hydroxide * 250 cm3 conical flask * 50 cm3 graduated cylinder * 2 mol dm-3 hydrochloric acid * Access to an ice bath (and clamps) * Vacuum filter (Buchner flask and filter) * Filter paper |
| **Safety factors** | Ensure risk assessments have been conducted and checked by an appropriate practitioner.  Reflux reactions involve heating glassware and reactants to high temperatures and can pose risk of burns if not monitored and handled correctly. Reflux apparatus should be left to cool before handling.  Safety goggles should be worn at all times whilst performing practicals and demonstrations.  Ethanol and methanol are flammable so should be kept away from naked flames.  Biodiesel is flammable and treat it as if it is corrosive and an irritant. |
| **CLEAPSS references** | Sodium hydroxide is corrosive, HC091a  Hydrochloric acid is corrosive, HC047a  Benzoic acid has organ toxicity and is corrosive, HC013A  Methanol is flammable, toxic and a health hazard, HC040b  Ethanol is flammable, an irritant and toxic, HC040A |
| **Prior learning** | * Completing the practical will be easier and will likely be speedier if students are familiar with the names of different glassware, have followed similar SOPs/methods previously, and have had experience putting together quick-fit (tight fitting) apparatus. |
| **Common misconceptions** | * Students sometimes are not confident with the naming of different glassware and will regularly set up a condenser wrongly by putting the water flow in the wrong way round. * When using jointed glassware, students are often unaware of the concept of positive/negative (male/female) joints and often try to connect glassware the wrong way around. * The differences when scaling up reactions is not an easy topic for students. It is important to note there may also be differences in safety considerations that students are not often aware of. * Many will not realise that the reaction temperature is limited by the solvent’s boiling point in a reflux, not by the temperature set on the heat source. Therefore, the choice of solvent is key when considering rate of reaction. For example, water will reflux hotter (100°C) and may therefore encourage a faster rate of reaction than a reaction in ethanol at reflux (78°C). |
| **Accessibility** | * Seek to ensure wide representation for any visiting speakers and case studies used (where appropriate). * The use of glassware and heating elements/Bunsen burners can be challenging for some students. Care needs to be taken when performing the practicals. * If there is not time/space/equipment to complete a full class practical, other options are provided. |

## Activity guide

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| **Introduction**  SUGGESTED TIME:  20 minutes  RESOURCES:   * L2 Slide deck – Slides 2–3 * L2 Teacher support sheet * Equipment outlined in L2 Teacher support sheet | * Go through the learning outcomes for this lesson with students on Slide 2. * Show students a demo reflux setup at the front of the class using the Teacher support sheet. Use a whole-class discussion to illicit the errors, any consequences or problems that may arise from leaving the setup as it is (safety issues, solvent/product loss, lower yield, etc.), and how the error can be corrected. As the errors are spotted and corrected, make those suggested changes to the setup. * Some setup errors which could be present in the equipment setup (as per Teacher support sheet) and some discussion points for consequences:   1. The condenser water inlet and outlet the wrong way around. (Correct would be in at the bottom, out at the top.) This can cause a decreased product yield due to solvent/reactant loss through the open top of the condenser as it is not efficient enough or cold enough to cool the vapours rising through it.   2. The lab jack flat on the desk or missing. (Correct would be raised off the desk.) This would mean when the reaction has finished and the heat source needs removing to allow it to cool: either wait until it has cooled completely before being able to move anything, or use heat proof gloves to handle the hot apparatus (and this could be unsafe).   3. A conical flask/pear-shaped flask used with a hotplate/poorly fitting heating mantle. (Correct would be a round-bottom flask with suitable heating mantle fitting snugly around it.) Conical flasks are rarely used for strong heating as they do not have a high surface area to volume ratio and the heating of their contents is very inefficient. Also discuss the use of a round-bottom flask (RBF) vs. using a pear-shaped flask and the considerations when choosing a flask: the heating mantle should fit snugly around the flask but not be too tight, the flask itself needs to be robust enough to undergo the reflux (RBF is often more robust than a pear-shaped flask), and a large surface area is needed for heating evenly (pear-shaped flask has a larger surface area to volume ratio for heating).   4. The setup is a closed system with a stopper in the top, either with or without a thermometer. (Correct would be an open system, no stopper in the top.) This would cause pressure build up as the reaction mixture vaporises into the gas phase and could cause an explosion.   5. No anti-bumping granules in the reaction vessel. (Correct is the use of a small number of anti-bumping granules.) This could mean the reaction mixture bumps and overspills, which is a safety concern and could cause loss of product yield.   6. Loose/incorrectly attached connection between RBF and condenser. (Correct would be close-fitting joints.) This can cause solvent/reactant loss through the gaps before it can reach the condenser.   7. An overfilled RBF. (Correct is less than half-filled.) This could cause a safety concern as the reaction mixture can bubble over, but also could cause solvent/reactant loss through the joints.   8. No clamp on the flask. (Correct would be a clamp on the flask.) This could cause a safety concern as the flask/condenser could fall over. |
| **Activity 1 – Reflux practical**  Suggested time:  55 minutes  Resources:   * L2 Slide deck – Slide 4 * L2 Worksheet * Equipment outlined in L2 Worksheet | * There are three options for practical activities to choose from on slide 4, which depend on the class’s ability, size, time and availability of equipment/chemicals. The choices include two full-class practical options, and the option of one microscale practical option. The student instructions are on the Worksheet. * Option 1: A full-class reflux practical to create biodiesel. This practical only needs to be left for 10 minutes to reflux so will be a quicker option. Students may need support in using an autopipette if one is used to dispense the methanol and/or sodium hydroxide. If there is time afterwards, a whole-class discussion can be used to compare mass yield results, and to discuss any errors which may have caused any discrepancies in yields. * Option 2: A full-class reflux practical to hydrolyse an ester, producing a carboxylic acid. This practical requires 30 minutes of reflux, so is a longer practical. There is a further option available to acidify and precipitate, and then perform a vacuum filtration of the product to isolate the impure solid product. The reflux time could be used to remind students how to perform or set up a filtration under vacuum, if needed. * Option 3: A microscale reflux practical. Consider this if there are limited space or resources. [www.linkedin.com/posts/bob-worley-12034631\_cleapss-activity-7157307723355684864-hHaQ/](http://www.linkedin.com/posts/bob-worley-12034631_cleapss-activity-7157307723355684864-hHaQ/) * After setting up the equipment, ask students to pause before continuing and ask another student to check their setup. This peer assessor should comment on what they have done correctly and inform them of any errors that need to be corrected. * Further options for videos to watch, and examples of reflux practicals can be found in the Industry links section of this Teaching Guide. If there are a few lessons available for this topic, the full synthesis of aspirin may be an interesting practical for students to complete, however it does involve more than just a reflux. (Further information about the synthesis of aspirin, its uses and methods for identifying it can be found at: <https://edu.rsc.org/resources/aspirin-book/56.article>) |
| **Plenary**  Suggested time:  15 minutes  Resources:   * L2 Slide deck – Slides 5–6 | * Ask students to discuss in pairs and create a short paragraph answering the scenario-based question on Slide 5. * Some points that could be given are:   + The condenser water inlet and outlet are the wrong way round meaning the condenser is not efficient enough to stop the escape and loss of gaseous solvents/reactants. Swapping the inlet and outlet will mean a greater cooling of reactant/solvent vapours leading to a higher return of reactants/solvent to the reaction vessel and reduced loss of reactants/solvent.   + The flow of water through the condenser is not high enough to provide sufficient cooling of the solvent/reactant vapours, meaning the condenser is not efficient enough to stop the escape and loss of gaseous solvents/reactants. Increasing the water flow through the condenser will mean a greater cooling of reactant/solvent vapours, leading to a higher return of reactants/solvent to the reaction vessel and reduced loss of reactants/solvent.   + The round-bottom flask (RBF)/reaction vessel used does not fit well into the heating mantle, meaning the reaction has not reached sufficient temperature, and the reaction has not gone to completion/the rate of reaction is not high enough. A closer/better fitting flask and heating mantle pairing will ensure more efficient heating and that the correct temperature is reached, leading to a more efficient refluxing process, greater reaction progress, a greater rate of reaction and a higher yield of product.   + The condenser is not attached properly, meaning solvent/reactant vapours are not returned to the reaction mixture or are lost to the environment, decreasing the rate of reaction and product yield. Ensure the condenser is attached to the reaction flask securely and with the condenser attached in a upright/vertical position above the flask. This will ensure any condensing reactants/solvents can drip directly back down into the reaction flask to continue the reaction.   + The reaction flask is overfilled, leading to loss of reactants/solvent through the joints/out the top of the condenser. Keeping the flask at maximum half-filled will mean it is less likely to spill over and cause loss of reactants/solvent, allowing for a more complete reaction and a greater product yield. * Revisit the learning objectives in Slide 6 to summarise the lesson before introducing the independent consolidation activity. |
| **Consolidation** (to be completed outside of lesson)  Suggested time:  20 minutes  Resources:   * L2 Slide deck – Slide 8 | * Ask students to research and write a short paragraph identifying and suggesting ways of improving the reflux process’s environmental footprint. * Some points could be: reducing energy consumption by using a solvent with a lower boiling point; introducing the use of a catalyst to reduce energy requirements; using a more efficient condenser; monitoring the solvent recycling to ensure the highest efficiency and reduced solvent/reactant loss; using heat exchange systems to recycle heat use; selecting a greener energy source for heat production; selection of a less hazardous/greener solvent with reduced disposal requirements. |

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| **Location** | **Link**  (with permission if required) | **Owner** | **Date last accessed** |
| Teaching Guide page 3 | <http://www.ncfe.org.uk/qualification-search/qualification-detail/t-level-technical-qualification-in-science-level-3-delivered-by-ncfe-883> (with permission) | NCFE\* | March 2025 |
| Teaching Guide page 4 | [www.technicaleducationnetworks.org.uk](http://www.technicaleducationnetworks.org.uk/) | Technical Education Networks | March 2025 |
| Teaching Guide page 6 | [support.tlevels.gov.uk/hc/en-gb/articles/360015345420-Industry-placement-logbook-for-students](https://support.tlevels.gov.uk/hc/en-gb/articles/360015345420-Industry-placement-logbook-for-students) | GOV UK | March 2025 |
| Teaching Guide page 7 | www.chemeurope.com/en/encyclopedia/Reflux.html | ChemEurope | March 2025 |
| Teaching Guide page 7 | [youtube.com/watch?v=mrD6dP3TLNw](https://www.youtube.com/watch?v=mrD6dP3TLNw) (with permission) | University of York/YouTube | March 2025 |
| Teaching Guide pages 7 and 14  Lesson 1 Slide 5 | [www.youtube.com/watch?v=\_SzYqPdUkFQ](http://www.youtube.com/watch?v=_SzYqPdUkFQ) (with permission) | Capilano University/YouTube | March 2025 |
| Teaching Guide page 7 | [www.youtube.com/watch?v=6VbNyFuYQMU](http://www.youtube.com/watch?v=6VbNyFuYQMU) (with permission) | ChemSurvival/YouTube | March 2025 |
| Teaching Guide page 7 | <https://chem.libretexts.org/Bookshelves/Organic_Chemistry/Organic_Chemistry_Lab_Techniques_(Nichols)/01%3A_General_Techniques/1.04%3A_Heating_and_Cooling_Methods/1.4K%3A_Reflux> | LibreTexts | March 2025 |
| Teaching Guide page 7 | [www.radleys.com/blog/reflux-reaction-mistakes-to-avoid/](http://www.radleys.com/blog/reflux-reaction-mistakes-to-avoid/) (with permission) | Radleys | March 2025 |
| Teaching Guide page 7 and 15 | <https://www.youtube.com/watch?v=hkBrw2fG75U> (with permission) | Cognito/YouTube | March 2025 |
| Teachers Guide page 8 | [edu.rsc.org/resources/interactive-lab-primer/1064.article](https://edu.rsc.org/resources/interactive-lab-primer/1064.article) (with permission) | Royal Society of Chemistry | March 2025 |
| Teachers Guide page 8 and 14 | [www.youtube.com/watch?v=aUEpAEaSsBU](http://www.youtube.com/watch?v=aUEpAEaSsBU) (with permission) | Malmesbury Education/YouTube | March 2025 |
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| Teaching Guide page 8 | [edu.rsc.org/practical/reflux-and-distillation-practical-videos-16-18-students/4012294.article](https://edu.rsc.org/practical/reflux-and-distillation-practical-videos-16-18-students/4012294.article) (with permission) | Royal Society of Chemistry | March 2025 |
| Teaching Guide page 8 | [edu.rsc.org/resources/the-preparation-of-2-hydroxybenzoic-acid-16-18-years/4017310.article](https://edu.rsc.org/resources/the-preparation-of-2-hydroxybenzoic-acid-16-18-years/4017310.article) (with permission) | Royal Society of Chemistry | March 2025 |
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| Teaching Guide page 8 and 22 | <https://www.linkedin.com/posts/bob-worley-12034631_cleapss-activity-7157307723355684864-hHaQ/>  (with permission) | Bob Worley/ LinkedIn | March 2025 |
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| Teaching Guide page 16  Lesson 1 Slide 14 | [www.chemtube3d.com/reflux-apparatus-360-interactive-exercise/](http://www.chemtube3d.com/reflux-apparatus-360-interactive-exercise/) | ChemTube3D | March 2025 |
| Teaching Guide page 22 | <https://edu.rsc.org/resources/aspirin-book/56.article> (with permission) | Royal Society of Chemistry | March 2025 |

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