



Perfumes with a pop: aroma chemistry with essential oils

Have fun with organic chemistry by extracting sweet-smelling essential oils from fragrant plants.

By Adrian Allan, Bob Worley and Mary Owen

For millennia, plants have been used for their medicinal benefits, flavours or smells. To determine which chemicals are responsible for certain properties in plants, scientists extract and analyse organic compounds in the laboratory. This can take many hours, so to enable students to try this

procedure in the classroom, we developed a method using an inexpensive microscale heater that extracts essential oils in a matter of minutes.

Using this heater, secondary school students can complete a number of distillation experiments to learn about the



chemical and physical properties of carbon compounds, including the effect of molecular polarity on solubility, and how to test for the presence of unsaturated carbon-carbon bonds.

Preparation: building the microscale heater

Teachers should build the microscale heater prior to the lesson. It takes 20–30 minutes and requires materials that can be found in most school science departments. We recommend that students carry out the activities in groups of 2–4, so several heaters will be needed.

Materials

For one microscale heater (figure 1):

- 10 cm x 4 cm piece of copper metal foil
- Heavy-duty scissors
- 7 ml glass vial with bung and glass delivery tube
- Elastic band
- 3 m of 0.45 mm (26SWG) enamelled (insulated) copper wire
- Matches
- Sandpaper or wire wool
- Mineral wool
- 30 ml jar (such as a mini jam jar)

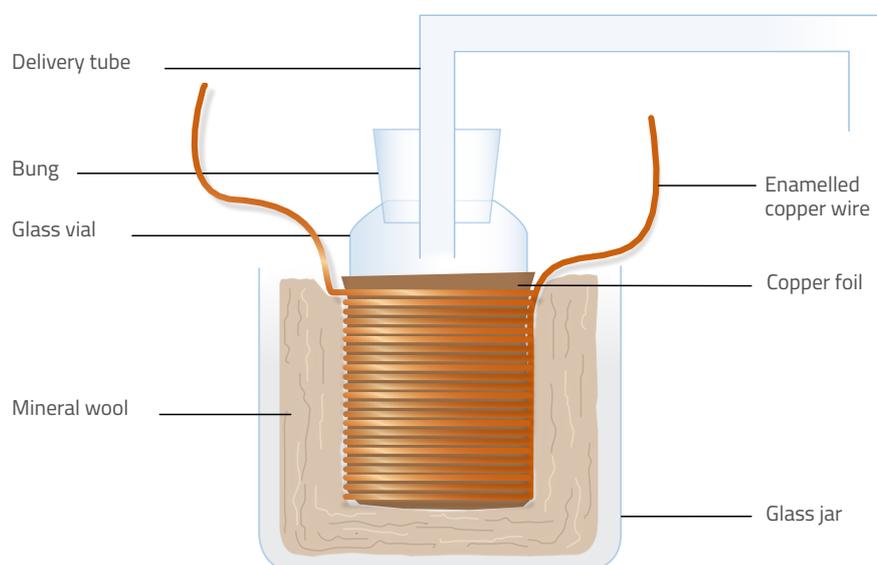


- ✓ Separation techniques
- ✓ Organic chemistry
- ✓ Aroma chemistry
- ✓ Ages 14–19

These hands-on experiments are ideal for introducing the topic of aroma chemistry and understanding how separation techniques are used to extract essential oils. The practical procedures are performed on a small scale, but are effective enough to illustrate large-scale industrial extractions that are usually carried out by steam or vacuum distillation. After distilling the essential oils, students can perform tests for unsaturation or reactivity, and discuss the theory behind them. A very interesting article!

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REVIEW



Adrian Allan/Nicola Graf

Figure 1: Diagram showing the elements of the microscale heater

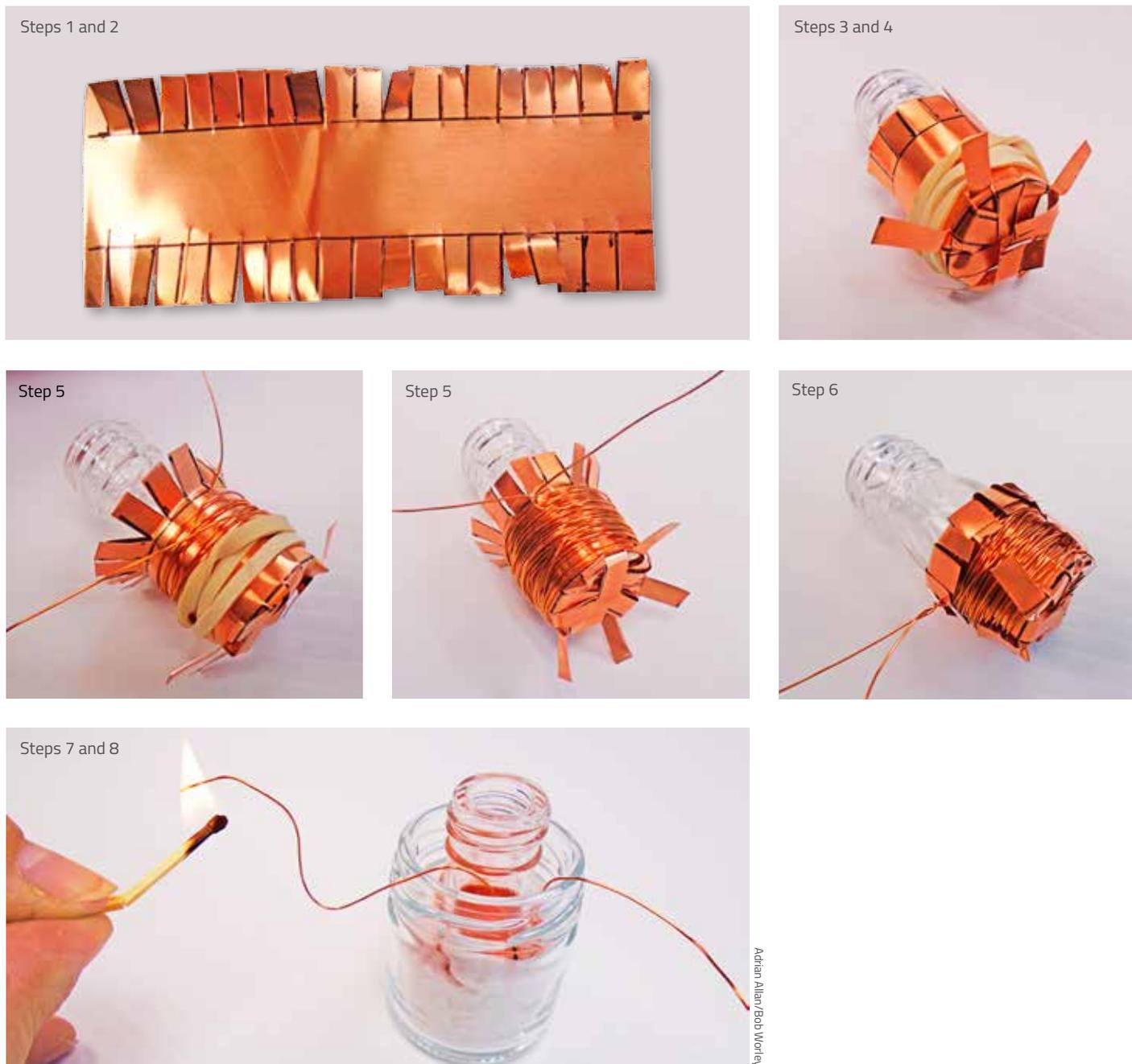


Figure 2: Steps for building a microscale heater

Procedure

1. Cut out a 10 cm x 4 cm piece of copper foil. Draw horizontal lines 1 cm from each long edge (figure 2). Draw vertical lines in 0.5 cm intervals.
2. Cut along the small vertical lines with heavy-duty scissors as far as the horizontal lines.
3. Wrap the foil loosely around the glass vial. Hold it in place temporarily using an elastic band.
4. Fold some of the bottom flaps of foil under the base of the bottle to hold it in place. Leave three or four flaps unfolded.
5. Wrap 3 m of enamelled copper wire around the copper foil, leaving about 8 cm of wire free at each end. The copper coil is your heating element.
6. Fold over the upper foil flaps and the remaining lower flaps to keep the wire coil in place.
7. Wrap the heater in mineral wool and place it in the jar with the loose wire ends sticking out.
8. Using a match, burn the ends of the wire to remove some of the enamel insulation. Rub the ends with sandpaper or wire wool to allow electrical contact. Ensure that the exposed wires are not touching each other, or the copper foil.
9. The glass vial can now be removed from the copper foil in preparation for activity 1.

Activity 1: Extracting essential oil using steam distillation

Essential oils are a mixture of volatile compounds that can be extracted from plants. They are composed mainly of a family of organic compounds called terpenes. In this activity, students use the microscale heater to extract essential oil distillates from orange peel, spearmint, lavender and cloves using steam distillation. The terpenes present in the oils are limonene, carvone, linalool and eugenol, respectively (figure 3). The activity takes about ten minutes. Each group of students will extract one type of essential oil.

Materials

- Microscale heater
- 7 ml glass vial with bung and glass delivery tube
- Plant material e.g. orange peel, spearmint, lavender and cloves
- Spatula
- 1–2 ml of distilled water
- Glass bottle (to collect distillate)
- Low-voltage DC power supply (able to deliver a current up to 6 A)
- Ammeter
- 2 crocodile clips and 2 leads (4 mm)

Procedure

1. Place two spatulas of plant material into the vial. This can be grated orange peel, dried leaves of lavender or spearmint, or a few dried cloves crushed with a pestle and mortar.
2. Add 1–2 ml of distilled water to the vial.
3. Insert the vial back into the copper wire coil of the heater, which is surrounded by mineral wool inside the jar.
4. Seal the vial by inserting the bung with a delivery tube. Place the other end of the delivery tube into a glass bottle to collect the distillate.
5. Using the crocodile clips, connect the ends of the copper wires to a low-voltage DC power supply (figure 4). A current of 4–5 A is required; an ammeter may be put in series to monitor this.

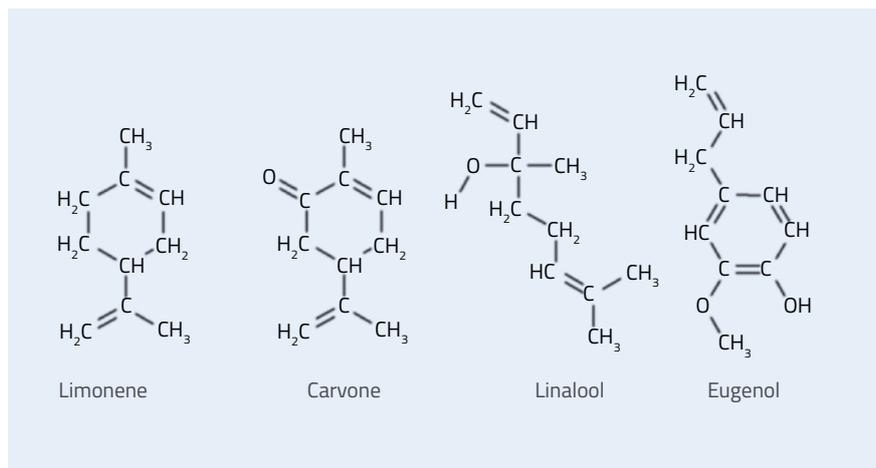


Figure 3: Example molecular structures of terpenes. From left to right, these chemicals are found in oranges, spearmint, lavender and cloves.

6. After 5–10 minutes, a colourless liquid will begin to drip into the bottle. Remove the first few drops of distillate from the bottle using a pipette for use in activity 2.
7. Continue heating the bottle until you have collected about 0.5 ml of liquid. This may take another 5–10 minutes.

- When terpene molecules evaporate, which forces are overcome: those between molecules or those within molecules?
- What compounds are present in the oil you have extracted?

Discussion

To explore the chemistry behind essential oils, discuss the following questions with your students:

- What is distillation? How can it be used to separate mixtures of substances?

Activity 2: Testing distillates for unsaturation

Terpenes – the primary component of essential oils – are unsaturated organic compounds, because they contain one or more carbon-carbon double bonds. This means they will react with bromine (in an addition reaction) or potassium



Figure 4: Experimental setup showing the microscale heater connected to the power supply

permanganate (in an oxidation reaction), resulting in the colour of the bromine solution changing from orange to colourless, and the potassium permanganate solution changing from pink to colourless. Limonene (the main terpene present in citrus fruits) can be used as an example (figure 5). This activity allows students to quickly demonstrate that unsaturated carbon-carbon bonds are present in essential oil extracts. The activity takes about five minutes.

Materials

- Distillates from activity 1
- Potassium permanganate solution: 0.002 M potassium permanganate (KMnO_4) in 0.1 M sulfuric acid (H_2SO_4)
- Aqueous bromine (Br_2) solution: 0.002 M
- Dropping pipette
- Spotting tile or laminated worksheet^{w1}
- Wooden splint cut to a point at one end, or cocktail stick

Safety note: Although the solutions are very dilute and used in small volumes, a lab coat and safety goggles are still recommended. If the solutions come into contact with skin, wash under the tap. Teachers should follow their local health and safety rules, in particular concerning the use of potassium permanganate and bromine and the disposal of the resulting solution. See also the general safety note on the *Science in School* website and at the end of this print issue.

Procedure

1. Using a dropping pipette, add one drop of potassium permanganate solution to two separate wells of a spotting tile. Alternatively, you can use a laminated worksheet with pre-labelled circles, which is available to print from the *Science in School* website^{w1} (figure 6). If using the worksheet, add the drops of solution to the two circles labelled 'potassium permanganate'.
2. Add one drop of bromine solution to two separate wells of the spotting tile, or the two worksheet circles labelled 'bromine solution'.
3. Add one drop of distillate (produced during activity 1) to one of the drops of potassium permanganate solution, and add another drop of distillate to one of the drops of bromine solution. Mix using a wooden splint. The distillate should decolourise the potassium permanganate and bromine solutions, showing that it contained unsaturated carbon-carbon bonds.

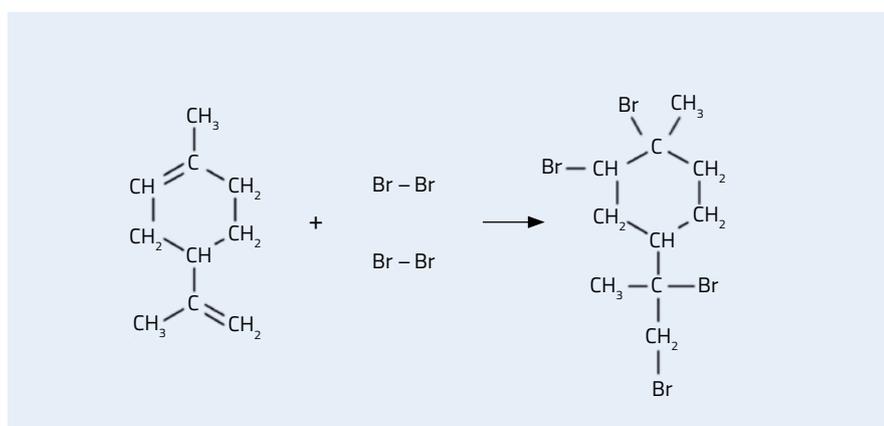


Figure 5: Molecular structures of the reaction of limonene with bromine

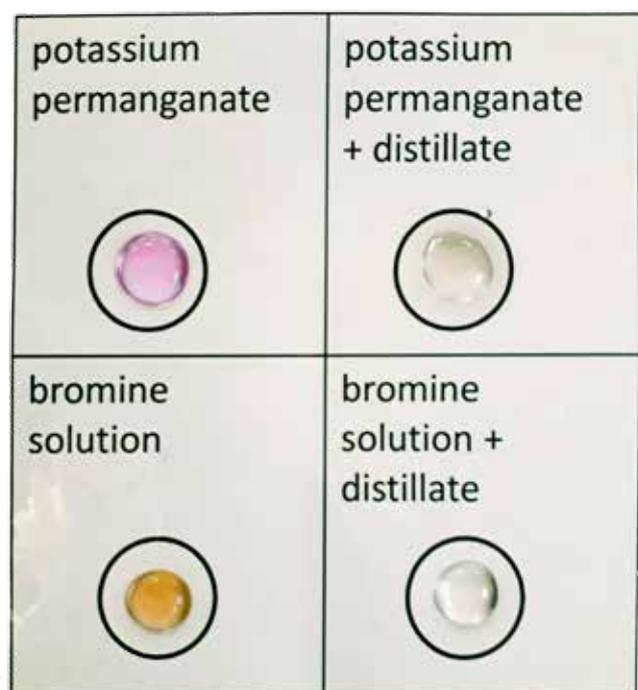


Figure 6: The essential oils decolourise potassium permanganate and bromine solutions, indicating the presence of unsaturated carbon-carbon bonds

Activity 3: Popping a latex balloon

Polar substances tend to dissolve in polar solvents, and non-polar substances may dissolve in non-polar solvents. This activity shows that latex can be broken up using a non-polar solvent (e.g. essential oils) but not polar solvents (e.g. water). When orange oil comes into contact with the balloon, the non-polar limonene molecules in the oil disrupt the weak forces between the polymer chains in latex to rupture the balloon and make it pop. The activity takes about five minutes.

Materials

- Distillation apparatus from activity 1 using orange peel
- Latex balloon
- Dropping pipette
- Tripod

Safety note: Check if any students or staff involved in the activity have an identified allergy to latex. If this is the case, take steps to limit exposure or in serious cases, do not carry out the activity.

Procedure

1. Move the distillation apparatus on top of a tripod, or allow the delivery tube to hang over a lab bench.
2. Inflate the latex balloon fully so that the rubber is stretched tight, and place underneath the delivery tube.
3. Start the distillation using orange peel, as in activity 1. The suspense will build as each drop of distillate falls from the delivery tube onto the balloon, until the balloon bursts^{v2} (figure 7).

Discussion

Encourage your students to think about the chemistry behind this fun trick by asking them the following questions:

- Why does limonene break up latex but water does not?
- Why is limonene used in many cleaning products?

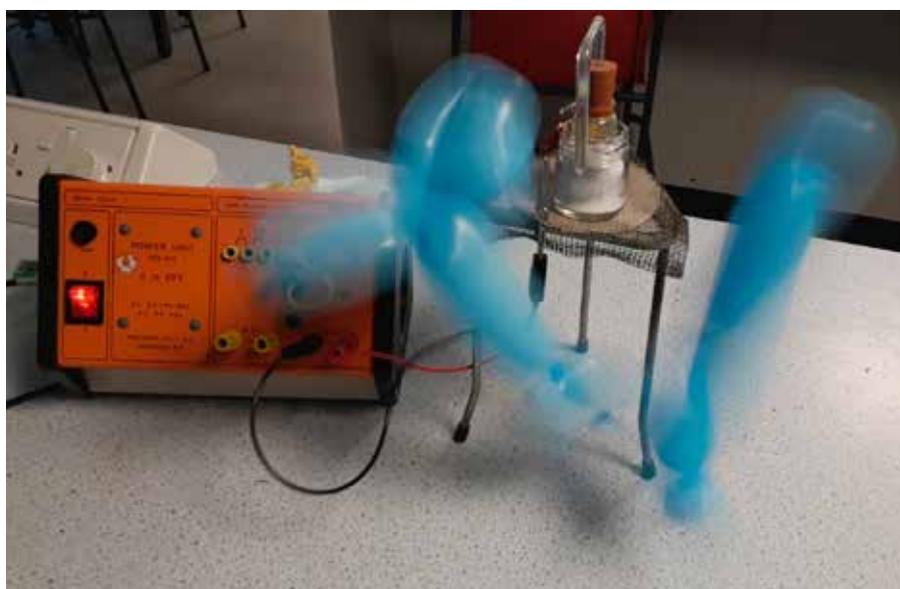
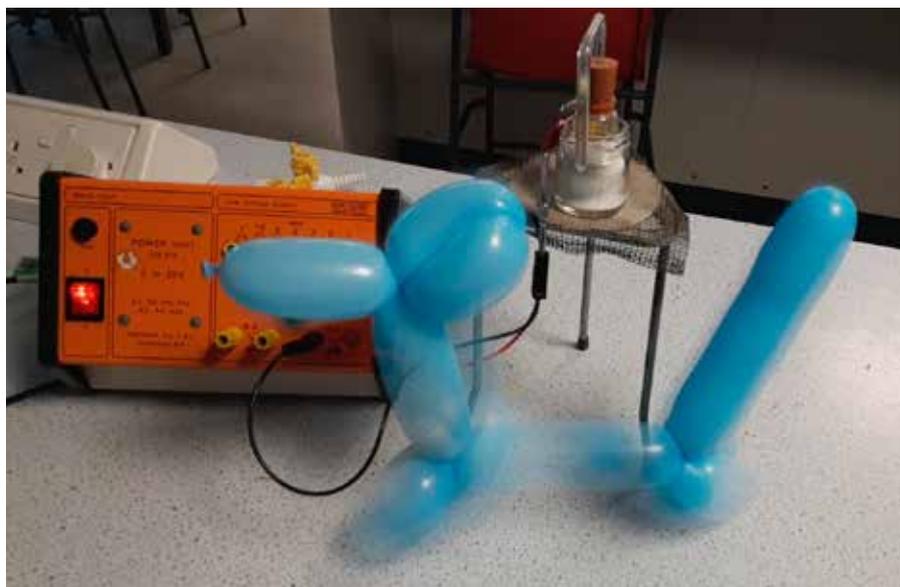
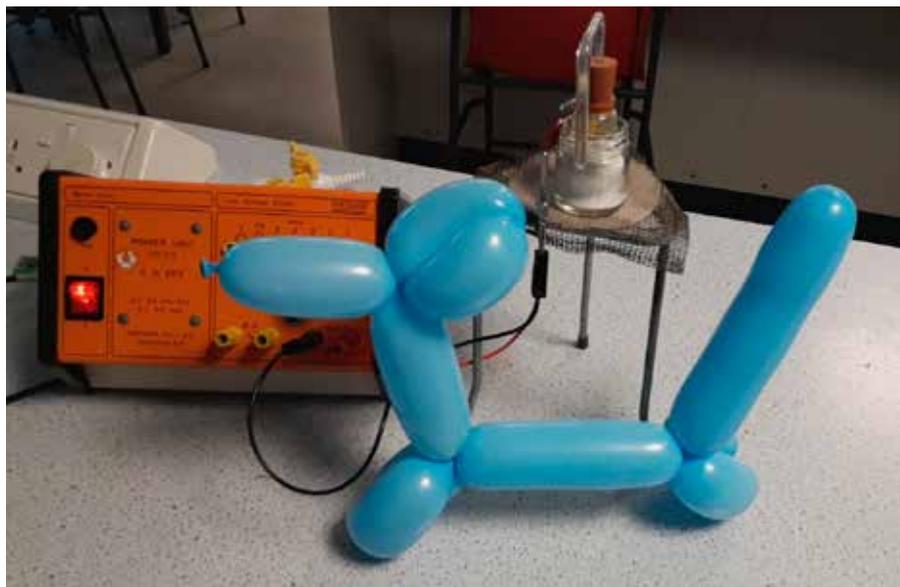


Figure 7: Limonene in orange oil dissolves the latex balloon, causing it to burst

Activity 4: Purifying essential oils

Students aged 16–19 can purify the essential oil by extracting it from the distillate using hexane. Hexane is a non-polar substance so it dissolves essential oil. The mixture of oil and hexane is then separated by evaporating hexane. This purification takes 10–20 minutes. Each group of students will need access to the following materials:

Materials

- Distillates from activity 1
- 3 glass Pasteur pipettes
- Test tube
- Small bottle of hexane (C₆H₁₄)
- Cotton wool
- Anhydrous sodium sulfate (Na₂SO₄)
- Wooden splint, cut in half lengthways
- Clamp and stand
- 7 ml glass vial
- Hot plate

Procedure

1. Add several drops of distillate to a small test tube using a glass Pasteur pipette. Add an equal volume of hexane and mix by bubbling air into the solution using the pipette.
2. Remove the lower aqueous layer with the pipette and discard.
3. Insert a small piece of cotton wool (2–3 mm in diameter) to the wider open end of an unused Pasteur pipette. Using half of the wooden splint, push the cotton wool down until it reaches the point where the pipette starts to narrow, just before the tip.
4. Using the splint, spoon some anhydrous sodium sulfate into the top of the pipette, enough for a few millimetres to rest on top of the wool.
5. Attach the pipette to a stand using a clamp and place a glass vial below it.
6. Transfer the remaining upper layer (a mixture of hexane and oil) to the top of the pipette. Sodium sulfate absorbs water molecules to form hydrates (it is a drying agent), so as the mixture passes through, any remaining water

will be removed. The cotton wool plug stops any solid sodium sulfate passing through with the mixture. The liquid will eventually enter the glass vial below. This will take 1–2 minutes.

If blockage occurs, attach a rubber teat and squeeze it to increase the air pressure inside the pipette. If this does not work, repeat the procedure using less cotton wool and sodium sulfate.

7. Place the glass vial on a hot plate in a fume hood and gently heat for 4–5 minutes until the hexane evaporates. A pleasant smelling oily residue will remain.

Discussion

During the extraction, discuss the following questions with your students:

- In the extraction, why was hexane added to the distillate?
- Why is the mixture of hexane and oil passed through anhydrous sodium sulfate?

As a follow-up activity, the extracted oil can be analysed using small-scale thin layer chromatography by following the procedure available on the *Science in School* website^{w1}.

Acknowledgements

The authors would like to thank Chris Lloyd for introducing them to using an electrical heater for distillation experiments in schools^{w3}, and Steve Jones, the director of CLEAPSS, for encouraging them to discover new techniques in microscale chemistry and for giving permission to use the microscale heater procedure in this article. They would also like to acknowledge Howard Tolliday and Hamish McGill for their efforts in developing and testing the procedures at Dornoch Academy, Scotland, UK.

Web references

- w1 The worksheet for activity 2 is available to download from the *Science in School* website, as is the procedure for analysing essential oils using thin-layer chromatography. See: www.scienceinschool.org/2018/issue44/distillation
- w2 A video showing the balloon demonstration is available on the *Science in School* website. See: www.scienceinschool.org/2018/issue44/distillation
- w3 Chris Lloyd from the Scottish Schools Education Research Centre (SSERC) developed his own method for building a microscale heater, which involved using plaster of Paris for insulation. Details can be found on the SSERC website. See: <http://info.sserc.org.uk/images/Chemistry/Chemistry%20Microscale/Making/HeatingMantle.docx>
- w4 Read about the Smells Like Teen Shampoo project in an article written by Adrian Allan's students, published in the *Young Scientists Journal*. See: <https://ysjournal.com/wp-content/uploads/2015/06/Smells-Like-Teen-Shampoo.pdf>

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