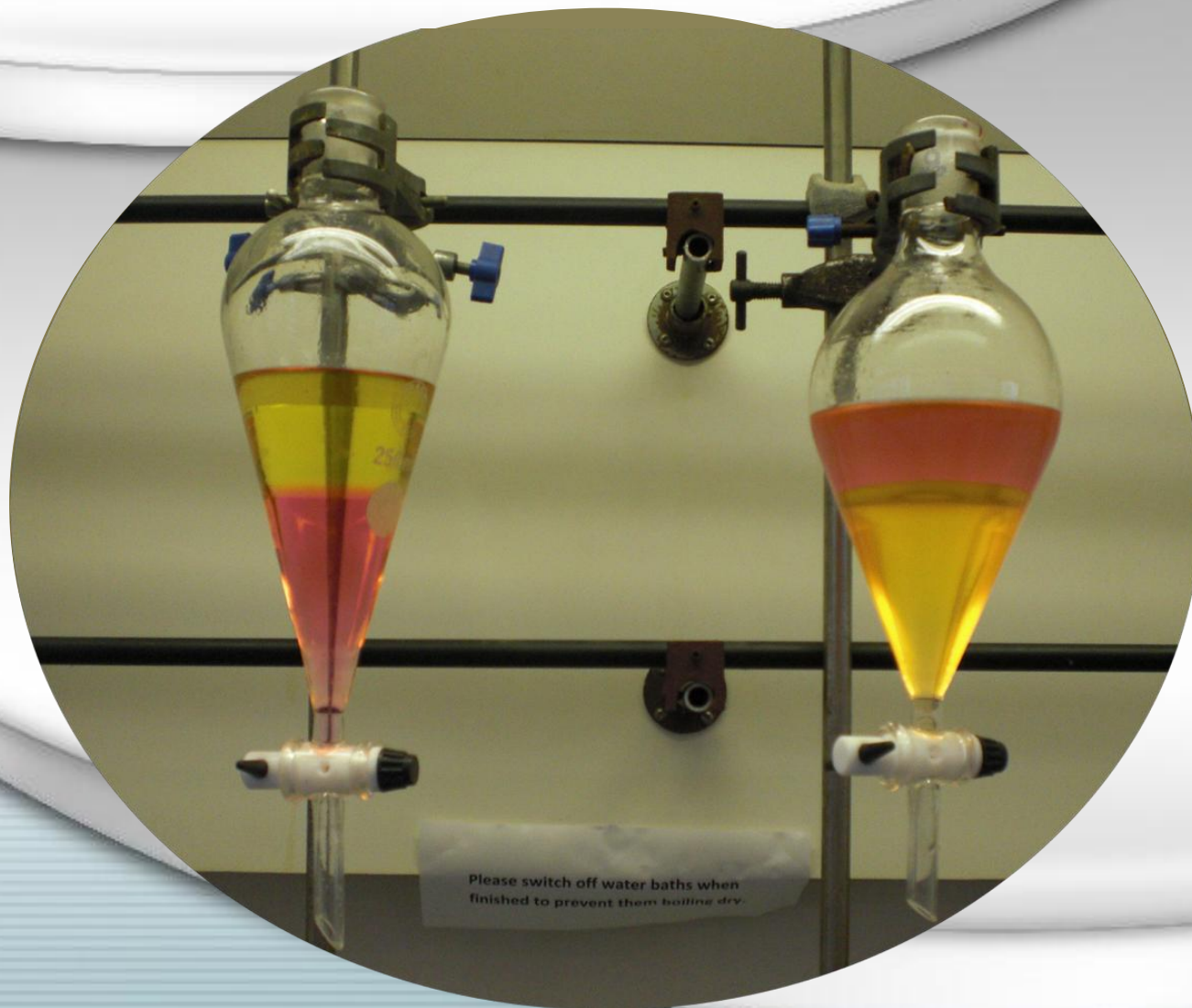


Using a separating funnel



Separating funnels are used for carrying out solvent extractions in organic chemistry. They can come in different shapes and volumes, make sure you use a suitable volume for your extraction and never fill the separating funnel more than two-thirds full.



When transferring liquids into the separating funnel you should make sure that the stopcock is closed and that the funnel is adequately supported, using a clamp or a ring.



When pouring liquids into the separating funnel you may wish to use a measuring cylinder or a long-stemmed glass funnel to minimise any spillages



Separating the layers is usually the part most find difficult. The main reason for this is not knowing which is the aqueous and organic layers. The table below lists a number of extraction solvents along with their relative densities:

Solvent	Density (g/cm³)
Petroleum ether	Typically ~ 0.640
Diethyl ether	0.714
Toluene	0.867
Ethyl acetate	0.900
Water	1.000
Dichloromethane	1.327

If your separation has gone well you should easily see two visible layers in the separating funnel. Here we have added colour to the aqueous and organic layers to show the separation more clearly, but for your separations there will be no colour.



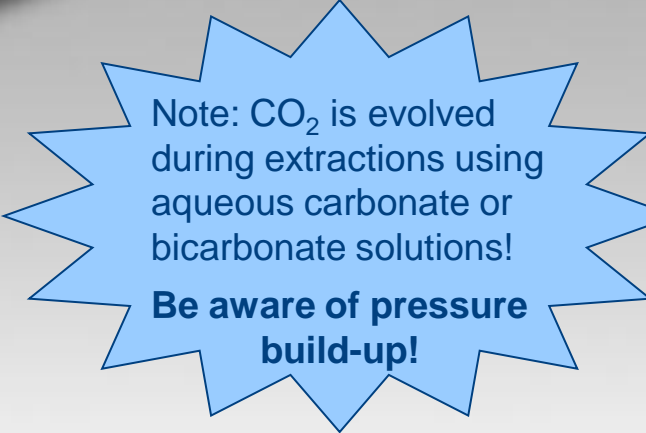
NOTE: In both separating funnels, the red layer is the aqueous layer. But how can this be?

Explanation: Look at the table on the previous slide. In the left separating funnel, the aqueous layer is on the bottom, meaning the organic layer must be less dense than water. In the right separating funnel, the aqueous layer is on the top, meaning the organic layer must be more dense than water.

Video 1

Screen capture walkthrough, with annotations

In order to carry out an efficient extraction the organic and aqueous layers must be thoroughly mixed. Here is a series of pictures that are screen captures of an accompanying video.



Note: CO₂ is evolved during extractions using aqueous carbonate or bicarbonate solutions!

Be aware of pressure build-up!

Video 1: Screen shots

1. Insert the stopper



2. Remove the separating funnel from the ring

Ensure solid grip is used when holding the separating funnel. One option is to put the stem between your forefinger and middle finger



Stem of flask

Video 1: Screen shots

3. Place one hand around the stopcock and keep the other over the stopper at all times



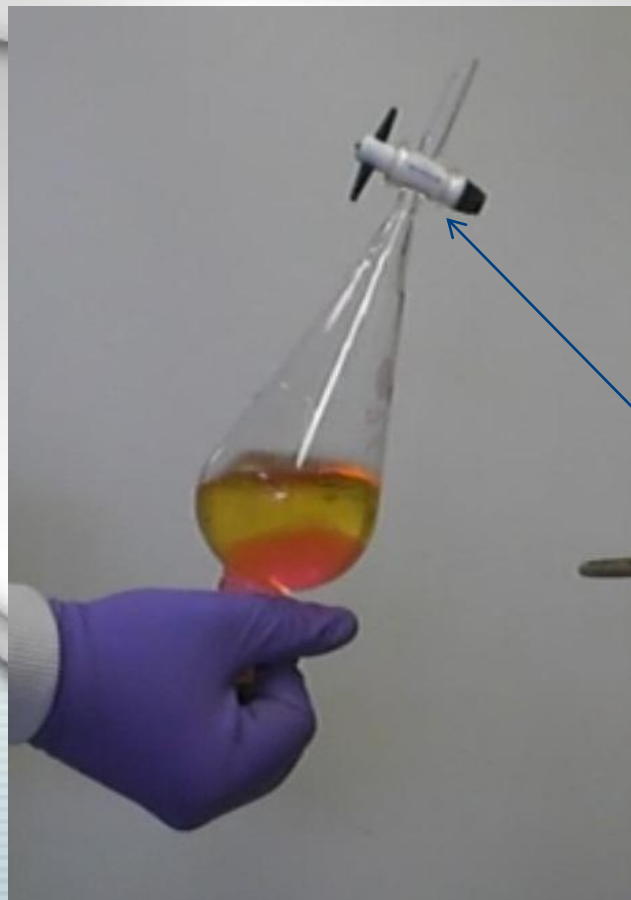
← Ensure the stopper is firmly in the palm of your hand that you have a good grip at the top of the flask

← To reiterate, ensure solid grip is used when holding the bottom of the separating funnel.

Stem of flask →

Video 1: Screen shots

4. Invert the funnel and immediately open the stopcock to release any pressure build up - **when venting a funnel never point the stem towards your neighbours or yourself**

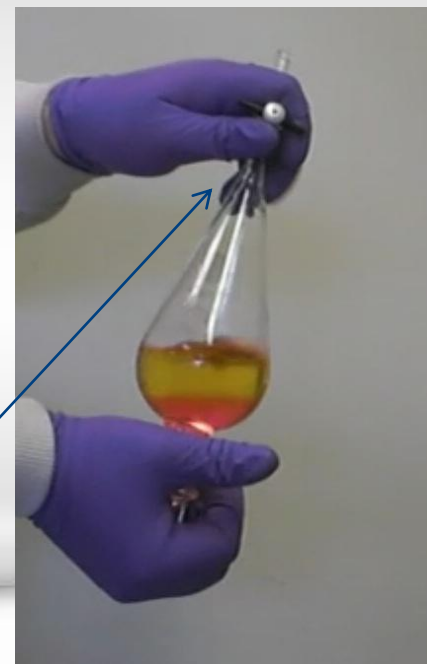


Note: Whilst not shown to the left (for ease of demonstration), always try and keep two hands on the separating funnel. This is shown properly in a few slides

Tap open: allows venting

Always ensure you close the tap before turning it the right way round!

5. Close the stopcock. Continued over page



Video 1: Screen shots

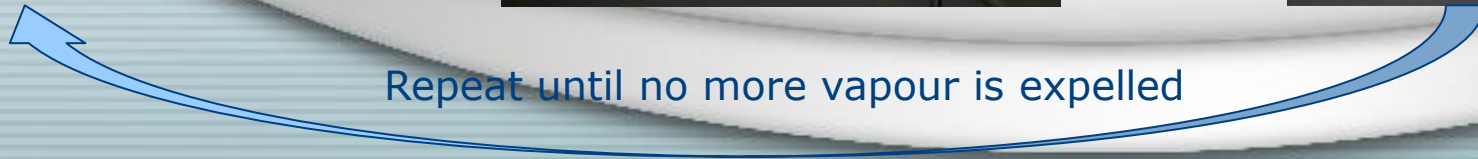
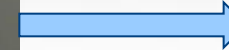
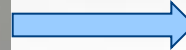
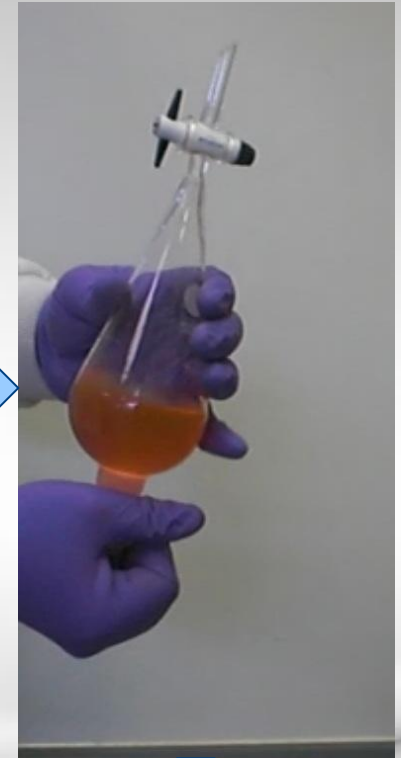
5. Close the stopcock and gently shake the mixture, release the pressure again by opening the stopcock
6. Repeat step 5 until no more vapour is expelled from the tap

Release pressure by venting. **Note** tap is open.

Close tap



Gently shake the mixture



Repeat until no more vapour is expelled

Video 1: Screen shots

7. Close the stopcock and return the separating funnel to the ring

8. Remove the stopper

Close tap



Return to retort ring



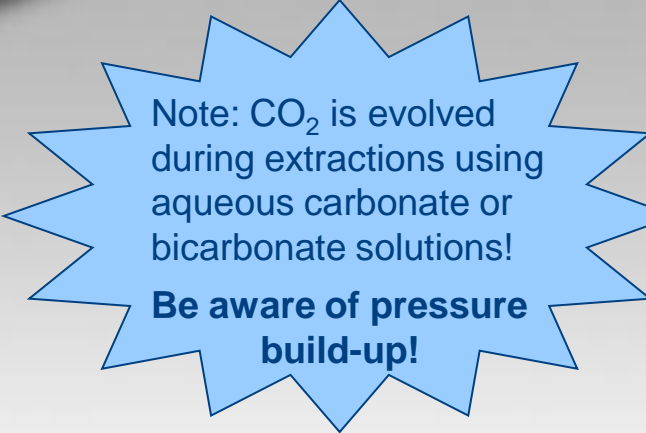
Remove stopper
(essential for
next step.)



Video 2

Screen capture walkthrough, with annotations.

Here is a series of pictures that are screen captures of an accompanying video.



Note: CO₂ is evolved during extractions using aqueous carbonate or bicarbonate solutions!

Be aware of pressure build-up!

It is now time to separate the layers into two different conical flasks, one for your aqueous layer and one for your organic layer (remember, colouring has been added here for emphasise – both your layers are likely to be colourless.

Note: This extraction is generic. You will need to determine whether you are extracting your aqueous layer with organic solvent, or vice versa. On this occasion, the red layer (bottom) is being extracted by the orange layer (top – called 'extraction solvent')

Video 2: Screen shots

1a. Separate your two layers into two different conical flasks

Remember: The layer being extracted does not necessarily have to be the bottom layer. It could also be the top layer

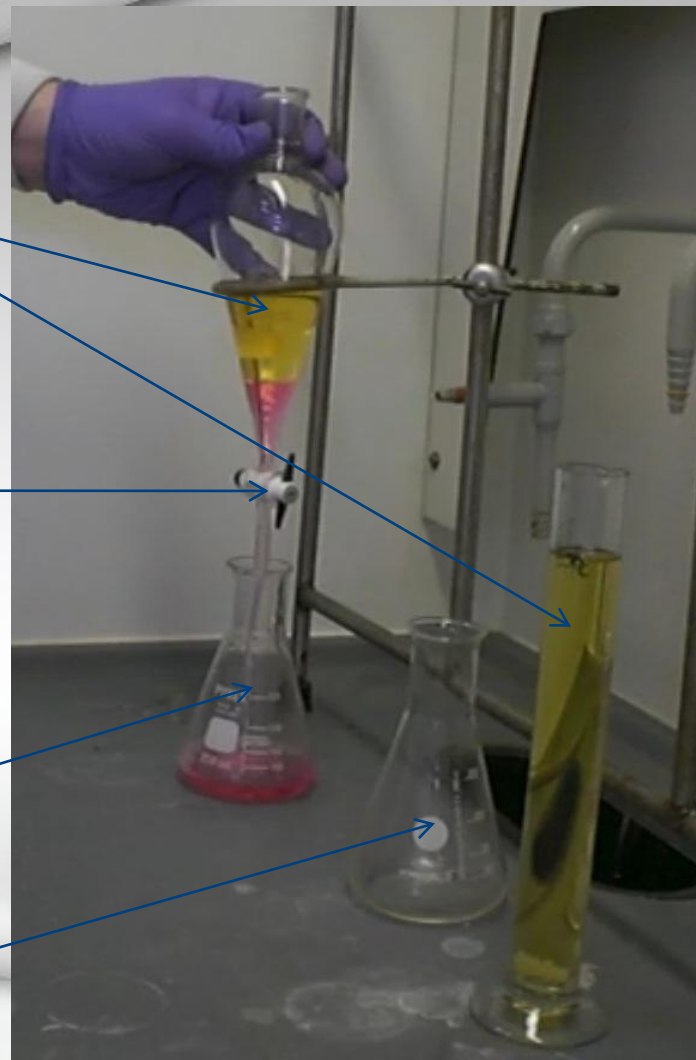


Layer being extracted

Extraction solvent

Open tap to run out bottom layer. Take care to keep layers separate.

Two different flasks. Make sure you label them properly (your solutions will be colourless!)



Video 2: Screen shots

1b. Separate your two layers into two different conical flasks

Close tap



Put empty flask under separating funnel and collect extraction solvent



Nicely separated



Video 2: Screen shots

2. Return the layer being extracted (red here) to the separating funnel (perhaps use a long stemmed funnel to minimise spillages)

Return the layer being extracted



Pour in 'new' extraction solvent from measuring cylinder



3. Add some more of your extraction solvent (orange), and repeat the shaking out process (see points 1-8 of video 1)

Back to similar situation as stage 1.

Note the amount in measuring cylinder has decreased



Video 2: Screen shots

4. After shaking and venting (steps 1-8 of previous video), separate your two layers again, you can combine this portion of extraction solvent (orange) with the previous extraction solvent (orange)

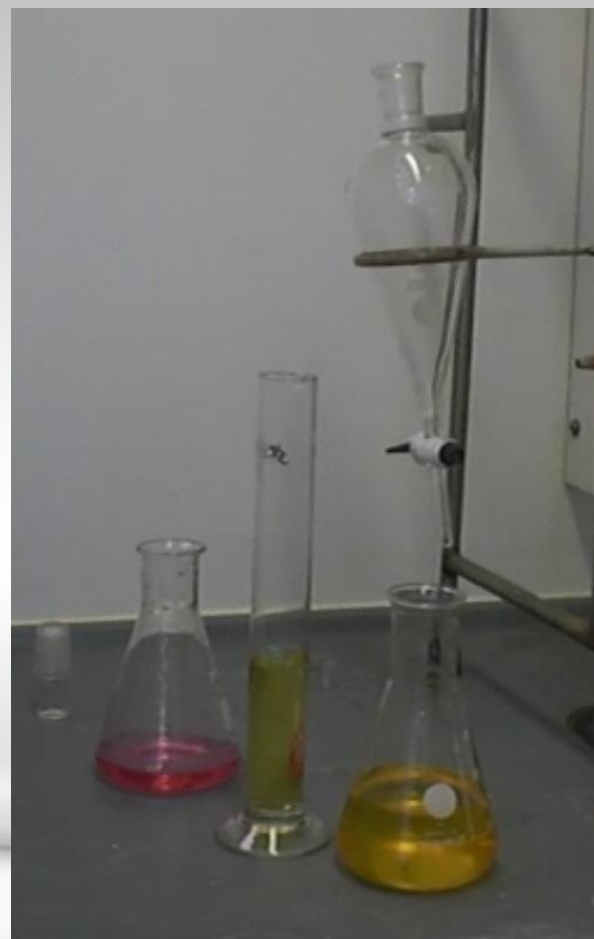
Remove layer being extracted into its original flask



Combine this portion of extraction solvent with previous portion



Nicely separated



Video 2: Screen shots

5. Repeat steps 2, 3 and 4 once more
6. You now have your two separate layers, make sure you know which one you need for further processing

Your extraction is finished. If the organic layer is the layer of interest (usually, but not exclusively), you should move onto drying the organic layer

