

Soxhlet extraction from leaves

Aims of the experiment

- To learn extraction as a purification process.
- To learn the Soxhlet apparatus as an extraction tool.
- Separation of mixtures of substances.
- To learn the partition law for a solid extraction.
- Preparing leaf extract.

Principles

Extraction is the name for a process in which a substance is removed from a solid, a liquid or a gas using an extractant. The material from which the substance is to be removed is also called the extraction material. At the end of the extraction, what remains is first of all the extract containing the desired substance and secondly the raffinate from which the substance was removed.

The extractant cannot be any arbitrary solvent. It may only remove the desired material from the extraction material, and must take up significant amounts of it. Also, the extractant must not chemically react with other materials contained in the extraction material.

Extraction is often used as a purification method when distillation or rectification cannot be performed. Areas of application for extraction are primarily found in the chemical industry. It is used to remove bauxite ore from iron oxide and to recover plant extracts in cosmetics.

A variant of extraction is the Soxhlet extraction process. This variant can remove sparingly soluble substances from solids by means of a solvent. Originally, this method was introduced by Franz von Soxhlet for determining the fat content in foods.

In this method the solvent is placed in a flask and is heated to boiling point. Then, solvent vapours rise in the entire apparatus and condense at a coil condenser. From here, the solvent drips onto a cellulose extraction thimble that sits in the Soxhlet extractor and which is filled with the extraction material. The extractant thus drips directly onto the extraction material and collects in the Soxhlet extractor. When a critical liquid level is reached, the Soxhlet extractor abruptly drains into the flask. This process is also called siphoning and is driven by way of a suction lifting effect. In the flask, the solvent has now been distilled from the extract and can thus remove new extract from the extraction material.

In this experiment, the Soxhlet apparatus is used in the extraction of leaves to remove leaf dyes from them.



Fig. 1: Set-up of the experiment.

Risk assessment

Ethanol is highly flammable. During the experiment, always make sure that the apparatus is sealed tightly and no fire sources are in the vicinity. The experiment should be carried out under a fume cupboard.

Ethanol	
	<p>Hazard statements</p> <p>H225 Highly flammable liquid and vapour.</p> <p>Safety statements</p> <p>P210 Keep away from heat/sparks /open flames/hot surfaces.</p>
<p>Signal word: Hazard</p>	

Equipment and chemicals

1	Coil condenser Dimroth, type 250 mm.....	665 422
1	Soxhlet extraction unit, 70 ml.....	665 453
1	Round-bottom flask, 250 ml, ST 19/26.....	664 301
1	Joint clip, plastic, ST 19/26	665 391ET10
1	Joint clip, plastic, ST 29/32	665 392ET10
1	Heating mantle, 250 ml, adjustable.....	666 6523
1	Laboratory stand 16 cm x 13 cm.....	300 76
1	Stand base V-shaped, large	300 01
1	Stand rod 75 cm, 12 mm diam.	300 43
2	Universal clamp 0...80 mm	666 555
2	Bosshead S.....	301 09
2	PVC tubing 7 mm diam., 1 m	667 193
2	Hose clamp 8...12 mm	604 460
1	Extraction thimbles 80 mm x 26 mm diam. .	661 050
1	Tweezers, blunt, 130 mm.....	667 027
1	Narrow-neck bottle, amber glass, 250 ml....	661 167
1	Funnel, Glass, 100 mm, diam.	665 005
1	Ethanol, denaturated, 1 L.....	671 9720
1	Stopcock grease, 60 g	661 082
1	Boiling stones 100 g.....	661 091
1	Glass wool, 500 g	672 1020
Additionally required:		
Green leaves, dry, finely crushed		

Set-up and preparation of the experiment

Set-up of the apparatus

1. The apparatus is set up as can be seen in Fig. 1.
2. Fix the stand rod in the stand base.
3. Attach two bosshead S clamps to the stand rods at a some distance apart, then fasten two universal clamps to them.
4. Fasten the 250 ml flask to the bottom universal clamp.

Note: Make sure that the height adjustment is such that the heating mantle and laboratory stand fit under the flask.

5. Place the Soxhlet extraction unit into the flask. To do so, rub a bit of grease onto the lower joint of the apparatus.
6. Place the coil condenser onto the Soxhlet unit. Grease the lower joint with stopcock grease as well for this purpose, and fasten using a joint clip.

7. Also secure the coil condenser using the second universal clamp.

8. To feed and return water, attach a tube to the two ends of the coil condenser using a hose clamp at each end.

9. Connect the tubes such that the water enters the spiral tube and leaves through the vertical tube. This creates counter-current cooling.

10. Place the laboratory stand below the flask and then the heating mantle on top.

Preparation of the experiment

1. Fill the flask about half way with ethanol and add some boiling stones.
2. Crush some of the dried leaves and place into the extraction sleeve so that it is about half full with them. Close the sleeve loosely with glass wool and place into the Soxhlet extractor.

Performing the experiment

1. Turn on the water cooling.
2. Turn on the heating mantle and raise the laboratory stand until the mantle surrounds the flask.
3. Carefully bring the ethanol to the boil.

Note: The ethanol should not boil too vigorously!

4. Allow the extraction to run for about 30 - 40 minutes.
5. The experiment is best ended when the liquid has been dispensed from the extraction apparatus for the second time.
6. Remove the heating mantle and allow the flask to cool.
7. The cooled extraction liquid can be kept in the dark in a brown glass bottle for further testing.

Observation

After a short heating time, the ethanol begins to boil and ethanol vapours slowly rise up in the apparatus. At the cooler, these vapours condense and drip into the extraction sleeve filled with the leaves, where they collect. Recondensed ethanol takes on a green colour after contact with the leaves. After the initial siphoning of the extraction liquid, a much greener ethanol residue remains in the extraction sleeve.

The ethanol in the flask also exhibits a greenish colour after the first siphoning, which intensifies significantly after the second pass.

Results

From the dried leaves substances were removed as an extract, which gave the ethanol its green colour. It is leaf dye, such as chlorophyll and carotenoids.

The extract obtained in this experiment can be further used for experiment C2.4.2.2.

Cleaning and disposal

Solvent residues must not be drained to the sewer. They must be disposed of in waste containers identified as organic solvent waste. Also, extraction material that is no longer needed must be disposed of as organic solvent waste. If the extract is used for further experiments, it should be kept in the dark if possible in a labelled brown glass bottle.