Purification of organic compounds

Advance Chemistry Lab 1

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Theory

Organic substances are diagnosed by measuring their physical constants, such as melting point, boiling point, refractive index, etc.

Melting point: is a specific temperature at which a substance transitions from a solid phase to a liquid phase. During this transition, the substance absorbs heat, causing its molecules or atoms to overcome the attractive forces that hold them in a rigid structure in the solid state. Once the substance reaches its melting point, further heating will result in a complete change of phase from solid to liquid.

The melting point is a characteristic property of a substance, and it can be influenced by factors such as pressure. In a broader sense, it provides valuable information about the physical behavior of a material and is widely used in various fields, including chemistry, material science, and quality control.

The melting point tends to have a narrow range for most pure compounds, and impurities can lower or broaden the melting range. Therefore, this phenomenon makes the melting point a crucial gauge for purity. It is important to record the melting range of an unknown substance to indicate its purity, as many pure organic compounds melt or decompose within a melting range not exceeding (1°C).

If the melting range is large, it may necessitate recrystallizing the substance in a suitable solvent, followed by re-measuring the melting point

Chemicals and Equipments

(1) Paraffin oil (2) Unknown melting point substance (3) Electric heater (4) Test tube, (5) Small cup (6) Thermometer (7) Rubber ring

It is preferable to use paraffin in the heating process for the following reasons:

1- Its specific heat is low, so its temperature can be changed easily, whether by heating or cooling.

2- Non-flammable when hot.

3- It does not cause corrosion, and due to its low specific heat, it does not cause severe burns when it falls on the hands.

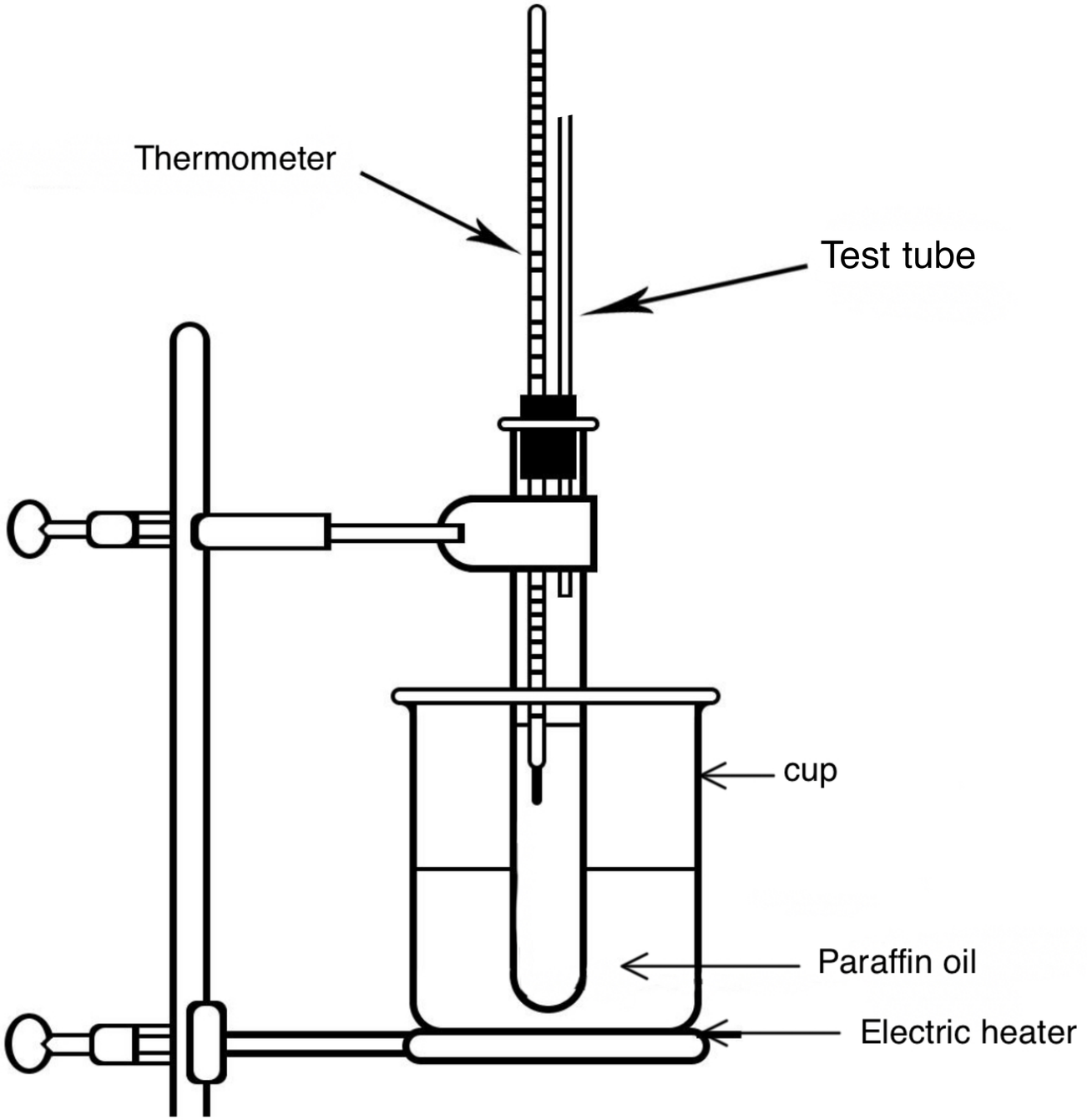
4- It can be heated to a temperature of (220°C) without any disintegration.

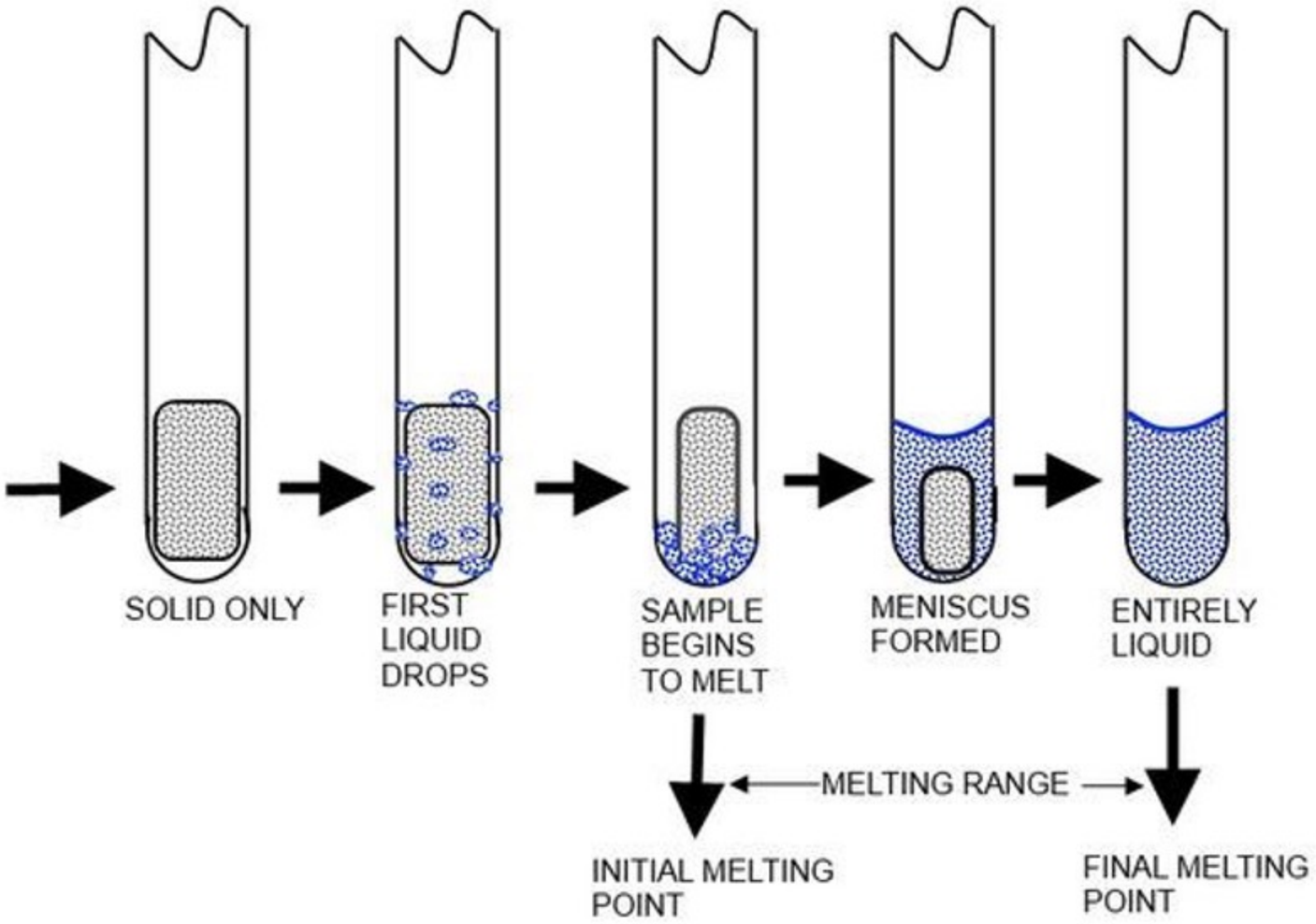
Procedures

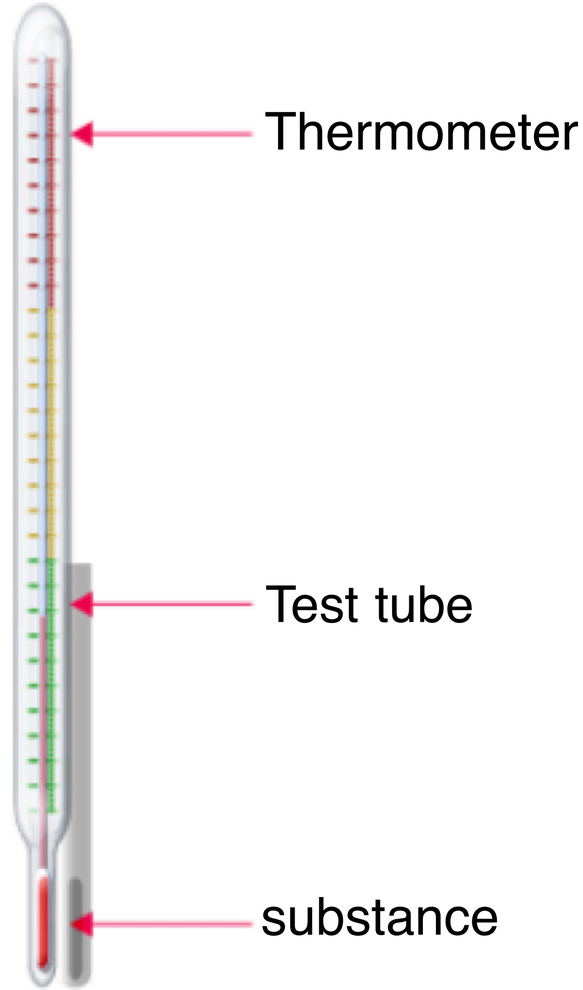
- Seal one end of an open-ended capillary tube by heating it.
- If the solid organic material is rough, crush it and insert it into the capillary tube. Repeat this process until the material at the tube's bottom measures approximately (1-2) mm.
- Attach the capillary tube to the thermometer using a rubber ring or a similar method, ensuring that the substance to determine the melting point is parallel to the heater's bulb.
- Place the capillary tube and thermometer in a small container (cup) with paraffin, filling it halfway with oil-paraffin mixture.
- Gradually heat the paraffin or oil bath with a low flame, ensuring the temperature rises no faster than 2-3 °C/min . Record the thermometer's pattern and temperature, noting the point at which the organic matter starts melting and its actual melting temperature.

The apparatus can be cooled and reused for subsequent experiments.

Note: Pure organic compounds typically exhibit a narrow melting point range, making it a reliable indicator of substance purity.

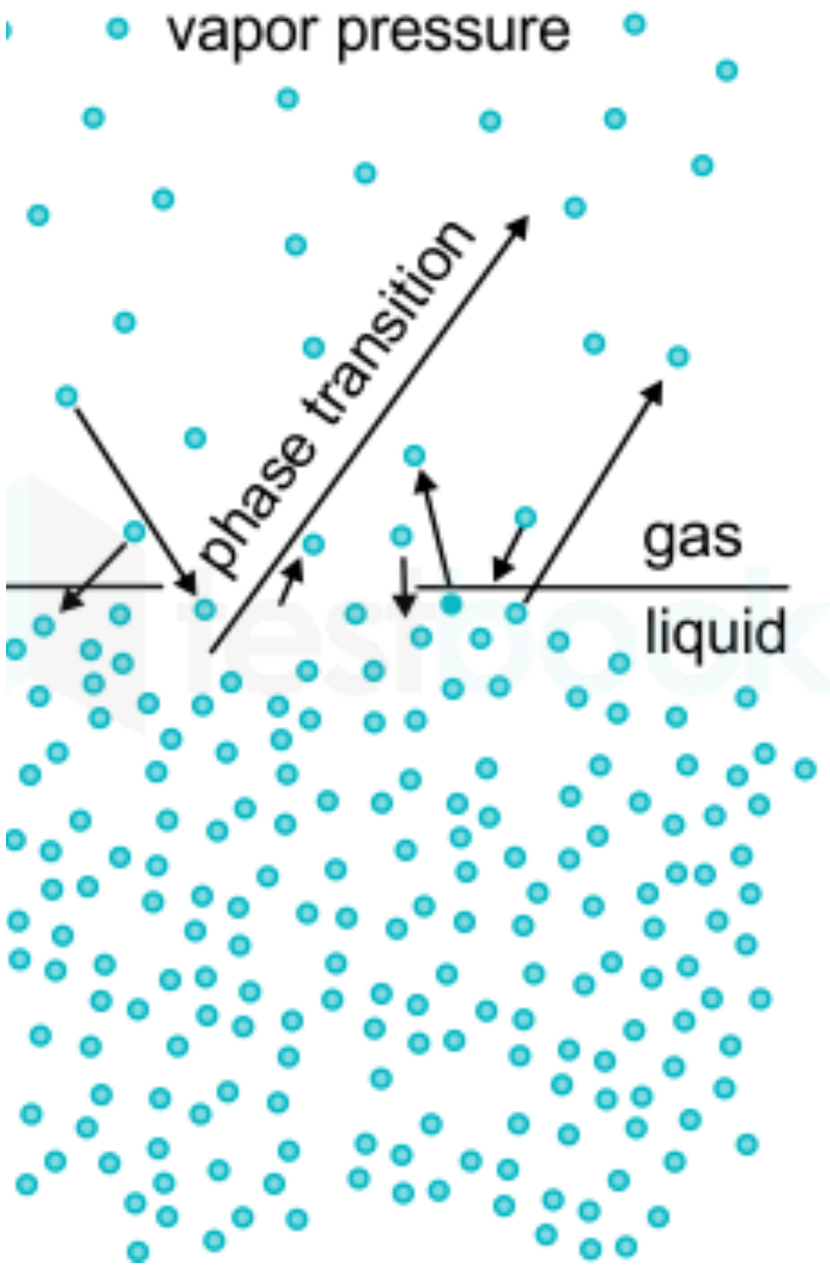



SOLID ONLY
FIRST LIQUID DROPS
SAMPLE BEGINS TO MELT
MENISCUS FORMED
ENTIRELY LIQUID
MELTING RANGE
INITIAL MELTING POINT
FINAL MELTING POINT


Thermometer
Test tube
substance

Boiling Point

The boiling point of an organic liquid is the temperature at which its vapour pressure equals the atmospheric pressure over the liquid leading to the transformation of the liquid into vapour. The boiling point is considered as a criterion of purity of a compound and is useful for identification of organic compounds. Similar to the melting point, the boiling point may be sharp or may vary over a temperature range. Pure liquids have sharp boiling points while mixtures show a boiling point range. It is a characteristic physical property that varies depending on the specific substance and the surrounding atmospheric pressure.

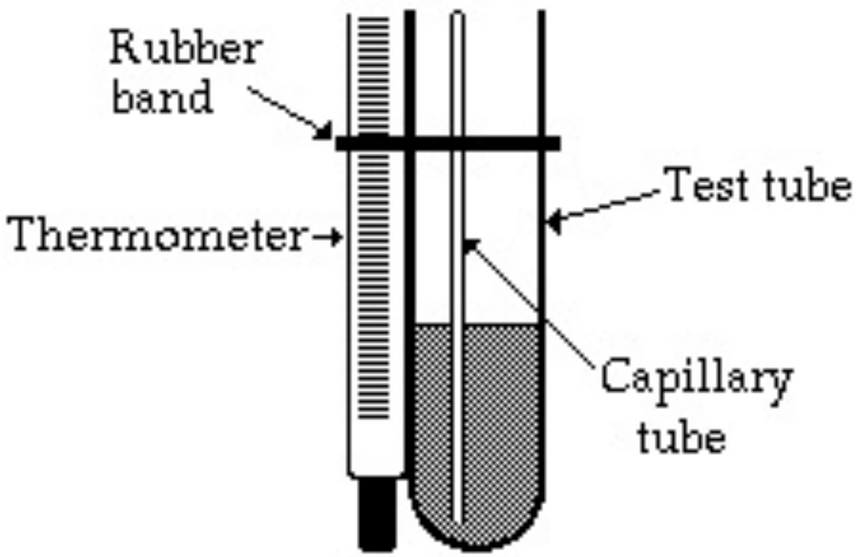


Chemicals and Equipments

(1) Paraffin oil (2) Unknown boiling point substance (3) Electric heater (4) Capillary tube, (5) Test tube (6) Small cup (7) Thermometer (8) Rubber ring

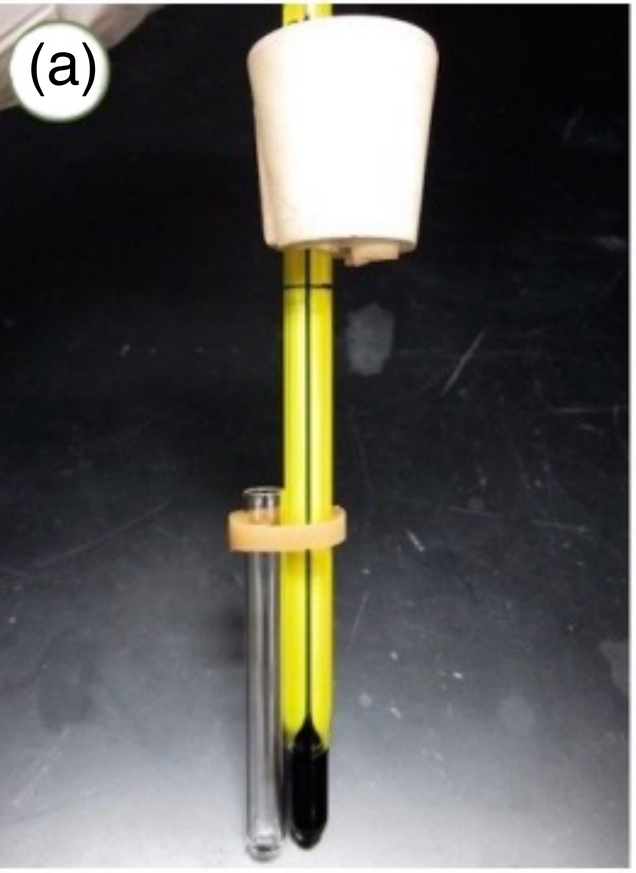
Procedures

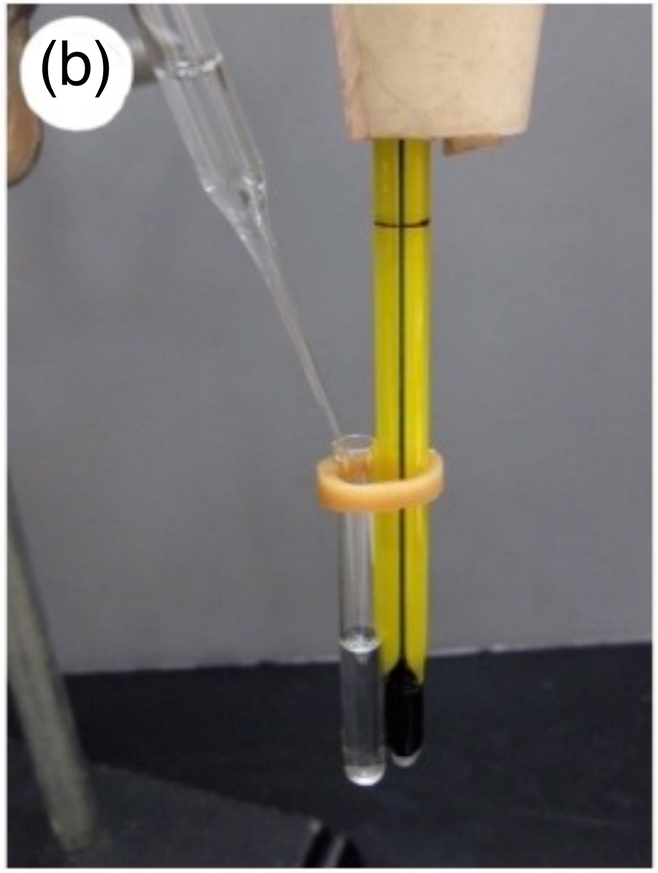
- Put about two or three drops of the unknown boiling point substance and put them in the test tube.
- Tie the test tube and the thermometer with the rubber ring, then insert the glass tube and the thermometer into the oil bath (paraffin). So that the rubber ring is above the surface of the paraffin.
- Turn the capillary tube inside the test tube after closing one end so that the closed end of the capillary tube is facing up and the open end is facing down.



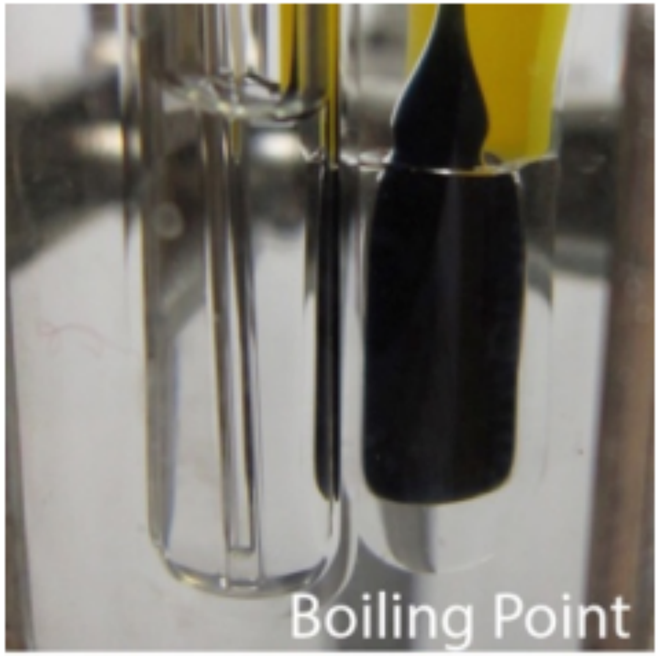
Boiling Point Apparatus

- When the oil bath start heating gradually, air bubbles will be released quickly and continuously from the end of the capillary tube immersed in the liquid. When this condition is reached, remove the heat source immediately and leave the device to cool. Note that the speed of the bubbles' exit decreases. At this moment when the bubbles stop coming out, record the temperature of the thermometer, which represents the boiling point of a liquid
- Remove the capillary tube from the test tube and allow the liquid that entered it to drain out. Then put it back in the tube after a certain period of time. The process is repeated using new drops of liquid. The goal of repeating is to measure the boiling point two or three times to ensure the correct degree. We will notice that the difference is one or two degrees Celsius at most.


(a)


(b)


(c)


Boiling Point

Purification of Organic Compounds by Recrystallization

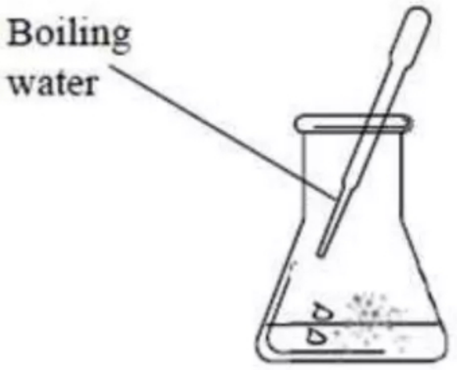
Recrystallization is a technique used to purify compounds by dissolving a mixture of a compound and impurities in an appropriate solvent.
Recrystallization is used to purify only solid organic compounds from impurities. This process is carried out by dissolving the impure raw material in the smallest possible amount of a suitable solvent while it is hot, then precipitating it in the form of crystals by cooling.

The recrystallization process can be summarized in the following steps:

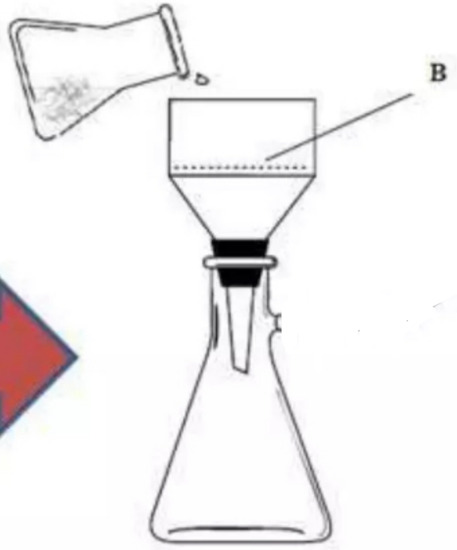
- Dissolving the compound to be purified in an appropriate solvent at its boiling point.
- Filter the hot solution to remove the suspended and insoluble materials and collect them on the first filter paper.
- Precipitation of the product (its crystallization) by cooling the filtrate resulting from the first filtration process.
- Filter the precipitate and collect it on a second filter paper to isolate it from the remaining solution after crystallization, which contains dissolved impurities.
- Dry the pure product.

The properties of the solvent used in crystallization process is:

- The substance to be purified must be dissolved in the solvent at its boiling temperature and precipitate in the form of crystals upon cooling.
- It does not interact with the compound to be purified.
- It is preferable that it be cheap, non-flammable, and non-toxic.
- It must not dissolve impurities or dissolve as little of them as possible
- Ability to produce large crystals of special shapes.
- Its boiling point is low so that it is easy to remove from the crystals of the pure compound during the drying process.



Step 1
Dissolve the impure Crystals
Just enough hot water to do the job



Step 2
Hot filtration with suction
Takes out non-soluble impurities

Step 3
Crystals allowed to reform
Recrystallisation

Step 4
Cold filtration under gravity
Takes out soluble impurities

Step 5
Dry the pure crystals