

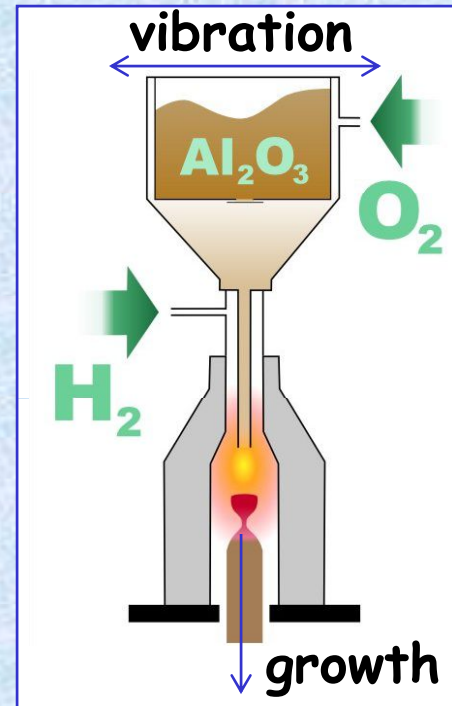
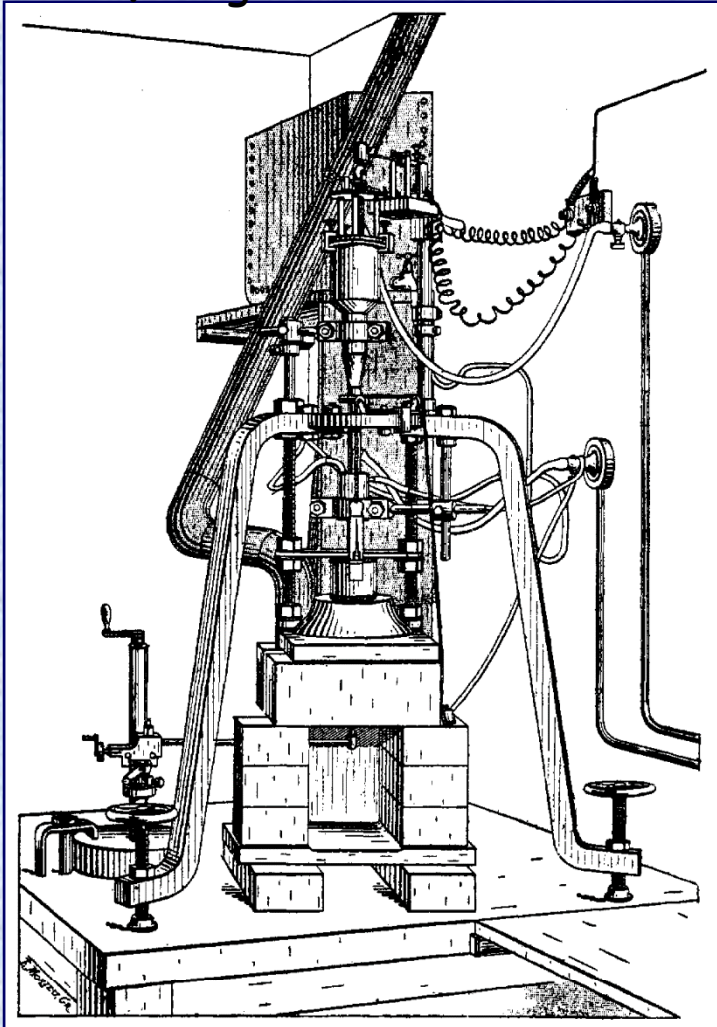
# **Verneuil method**

The **Verneuil process**, also called **flame fusion**, is a method of manufacturing synthetic gemstones, developed in 1902 by the French chemist Auguste Verneuil. It is primarily used to produce the ruby and sapphire varieties of corundum, as well as the diamond and strontium titanate.

- The principle of the process involves melting a finely powdered substance using an oxyhydrogen flame, and crystallising the melted droplets into a boule. The process is considered to be the founding step of modern industrial crystal growth technology, and remains in wide use to this day.

# Verneuil

1902, Auguste Verneuil



# Verneuil Process

- First used ~1902-1910 for large-scale sapphire / ruby growth ( $\text{Al}_2\text{O}_3$ )

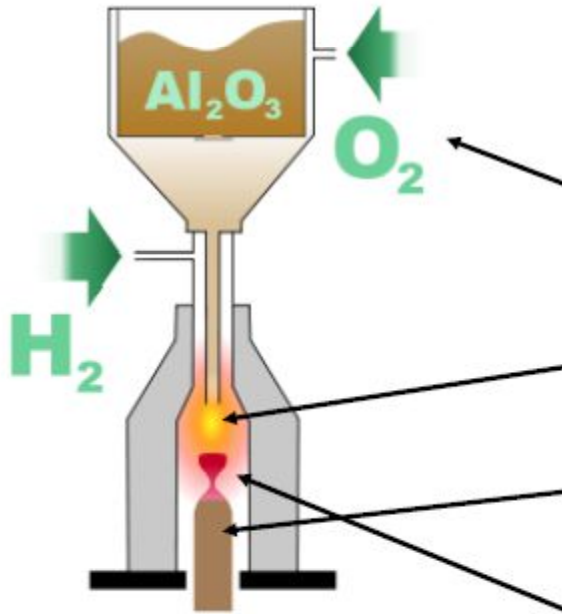
$\text{O}_2 + \text{Al}_2\text{O}_3$  inlet

$\text{O}_2 + \text{H}_2$  mix and ignite,  $T > 2000\text{K}$

Molten drops fall onto “pedestal”

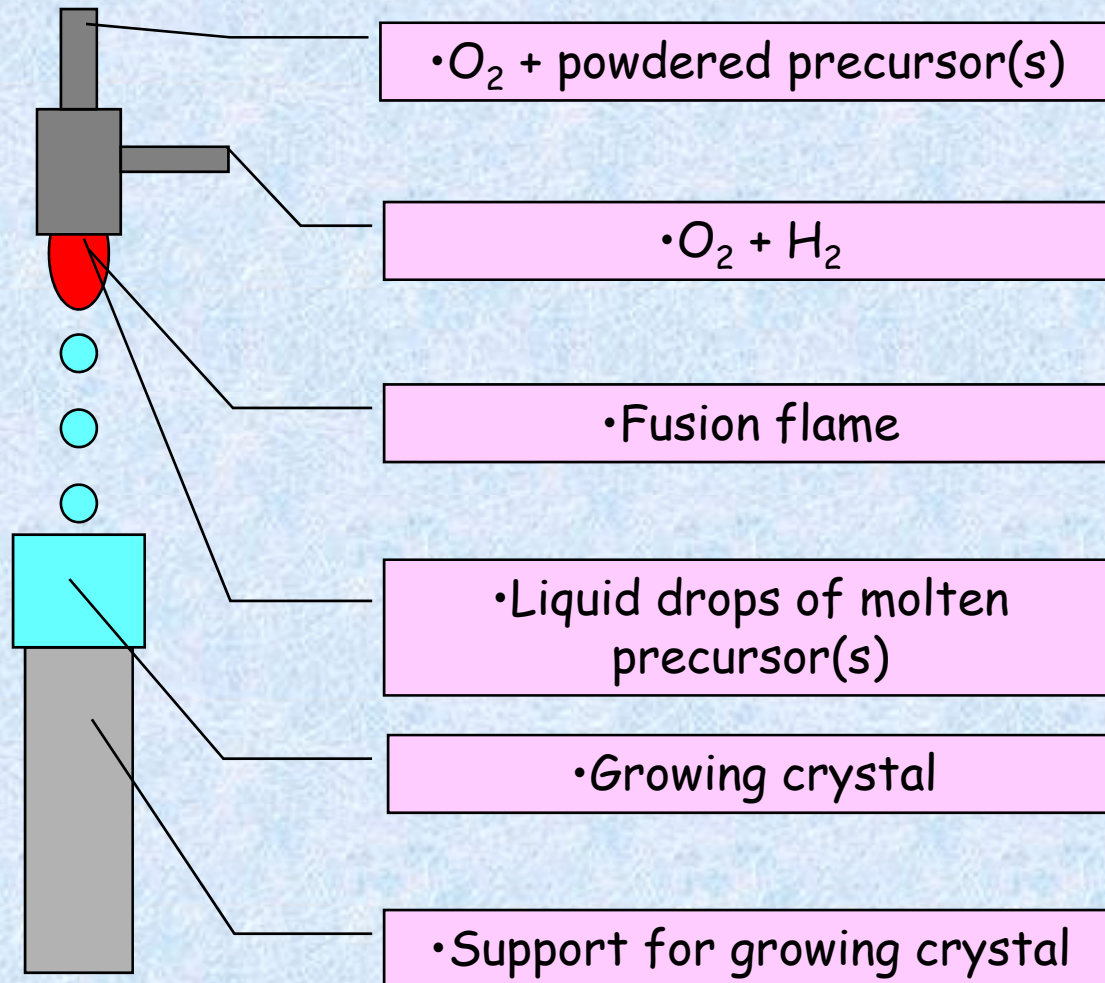
xtal forms & grows

Example of  $\text{Al}_2\text{O}_3$  xtal (right end)



- The process was designed primarily for the synthesis of rubies, which became the first gemstones to be synthetically produced
- One of the most crucial factors in successfully crystallising an artificial gemstone is obtaining highly pure starting material, with at least 99.9995% purity. In the case of manufacturing rubies or sapphires, this material is alumina

## •VERNEUIL FUSION FLAME METHOD



- This starting material is finely powdered, and placed in a container within a Verneuil furnace, with an opening at the bottom through which the powder can escape when the container is vibrated. While the powder is being released, **oxygen** is supplied into the furnace, and travels with the powder down a narrow tube. This tube is located within a larger tube, into which **hydrogen** is supplied.



- At the point where the narrow tube opens into the larger one, **combustion** occurs, with a flame of at least 2000 °C (3,600 °F) at its core. As the powder passes through the flame, it melts into small droplets, which fall onto an earthen support rod placed below. The droplets gradually form a **sinter** cone on the rod, the tip of which is close enough to the core to remain liquid. It is at that tip that the **seed crystal** eventually forms. As more droplets fall onto the tip, a **single crystal**, called a *boule*, starts to form, and the support is slowly moved downward, allowing the base of the boule to crystallise,

# SOLUTION GROWTH METHOD

**1. Crystal Growth by Slow Evaporation of the Solvent**

**2. Crystal Growth by Slow Cooling of the Solvent**

# **1. Crystal Growth by Slow Evaporation of the Solvent**

**Substances that are moderately soluble at room temperature, and which are either not much more soluble or are unstable at higher temperatures are good candidates for crystal growth by slow evaporation.**

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- Prepare a saturated solution, filter it, and place it in a beaker. Cover the beaker with foil or wax into which a few small holes have been punched. 2-3 pin-holes in parafilm over a 10 mL beaker works well, as long as the solvent does not dissolve the parafilm. Aluminum foil may be used if parafilm is a problem. - Check the solution each day. It is important to harvest the crystals before all of the solvent has evaporated

## **2. Crystal Growth by Slow Cooling of the Solvent**

**Substances that are much more soluble in a solvent at high temperature than at low temperature are good candidates for crystal growth by slow cooling.**

Make a saturated, or nearly saturated, solution just below the boiling point of the solvent. Filter while hot if any solid remains. Allow the solution to cool slowly. If the solution cools too quickly, owing to the coolness of the room, you may surround the beaker with insulation. Commercial ovens are available, but a Styrofoam cup or packing base from acid bottles covered with metal foil may work as well to slow the rate of cooling.

# Steps involves in solution growth

- Making a saturated solution
- Growing seed crystals
- Growing a crystal

*Crystal growing is like meditation. A calm and steady environment produces healthy crystals. Any variety of disturbances will lead to imperfection and discord.*



## Making Seed Crystals

Find a wide low jar. Clean it well and pour some of the saturated solution into it. Cover the low jar with a clean piece of cloth; this will allow the water to evaporate and keep dust from falling into the jar.

Set the jar in a dark area and wait. Gradually, as the water evaporates, tiny crystals will begin to grow at the bottom of the jar. Eventually, these crystals will be large enough to work with using your fingers. Remove these "seed crystals" from the jar. They will be used to make much larger crystals in the next step.

Sometimes, instead of getting a few seed crystals growing, you get a whole carpet of tiny crystals growing on the bottom of the jar. This means either the water or the jar was dusty.

## Making a Saturated Solution

The first step in making crystals involves preparing a water solution that has as much alum dissolved in it as possible. This is called a saturated solution.

Start with a clean jar that can be covered and shaken. Add water to the jar.

Add an amount of alum to the water. Shake well and let stand for 30 minutes.

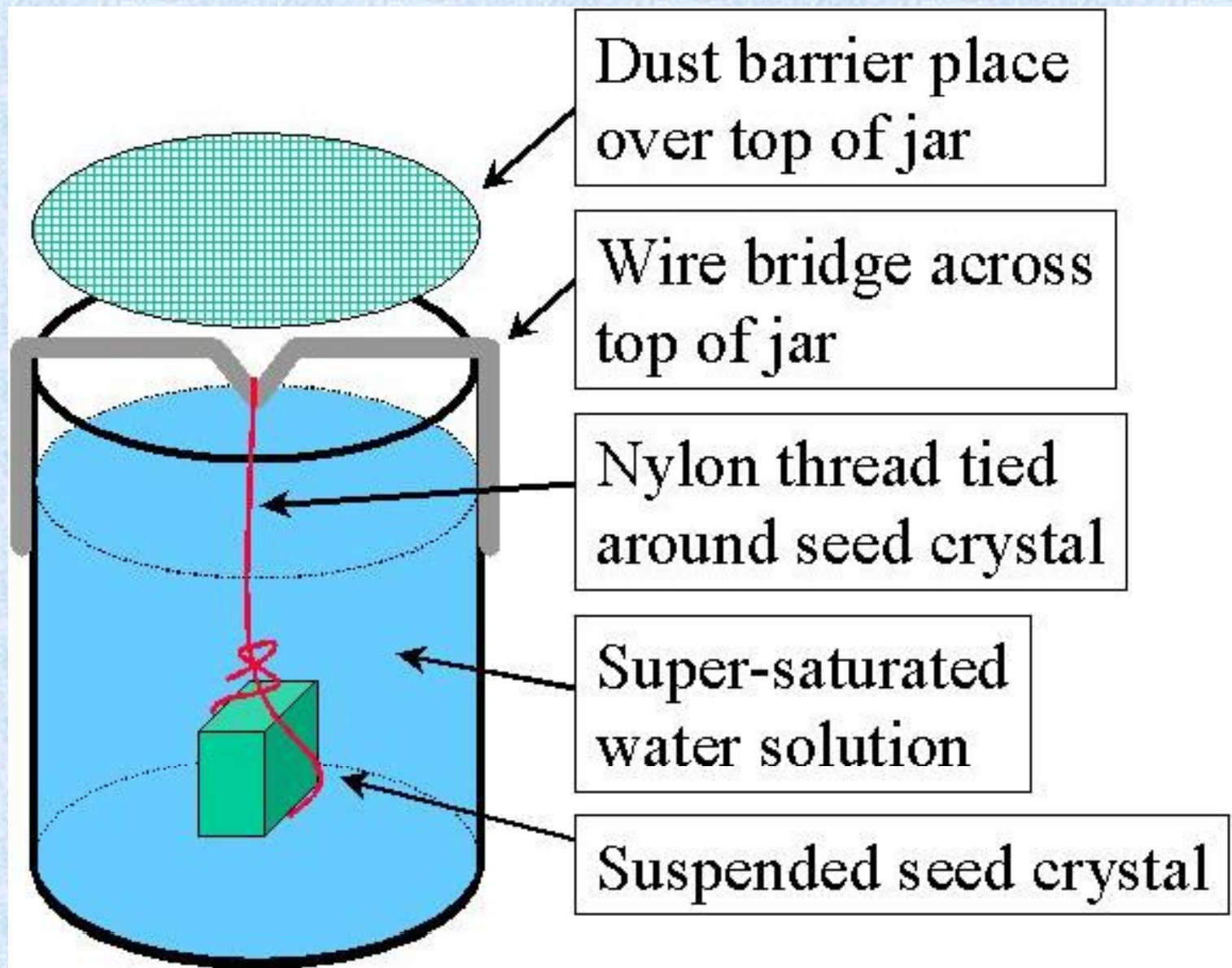
If there is NO powdered alum visible on the bottom of the jar then repeat step 2 until there is.

Carefully pour off the liquid into another clean jar (without getting any powder into the liquid) and seal this new jar tightly. This is your saturated solution.

# **Mason jar method**

(slow evaporation  
method)

This method was  
introduced by MASON  
AND SINGER IN 1960



## Growing Crystals

Clean a wide jar and place a wire over it as shown in the figure below.

Fill the jar with saturated solution.

Using thin nylon thread (or fishing line), tie a string to the seed crystal. You may need to scratch some small grooves into the seed crystal for the string to hold onto the seed. Avoid fabric threads since tiny seed crystals will form along the lint ends of the thread. The result will be a gumble of crystals and not a single large crystal.

Suspend the seed crystal into the saturated solution close to the bottom of the jar. Use a piece of tape to fix the string to the side of the jar.

Cover the top of the jar with clean cloth. This keeps dust from falling into the jar and allows the water to evaporate out



Finally, place the jar in a spot out of the sun where the temperature of the air does not change over time. Gradually, over time, the water in the jar will evaporate. Solid alum will leave the saturated solution and deposit onto the seed crystal. The crystal will grow and change shape. Because the crystal grows faster in some directions and slower in other directions, the crystal doesn't look round like a ball. **The crystal's shape is determined by the directions that grow the slowest.**

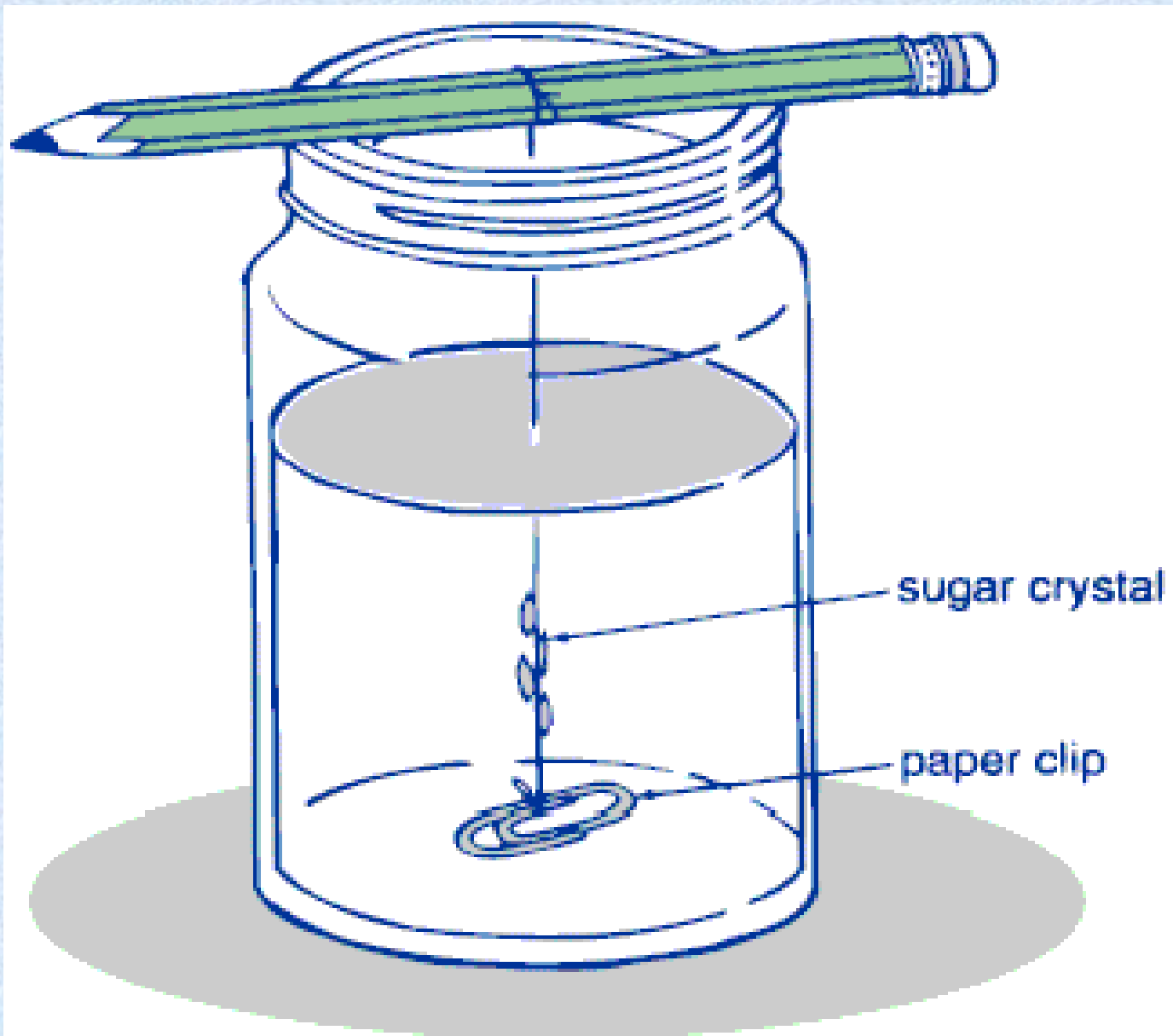


Figure 25.2

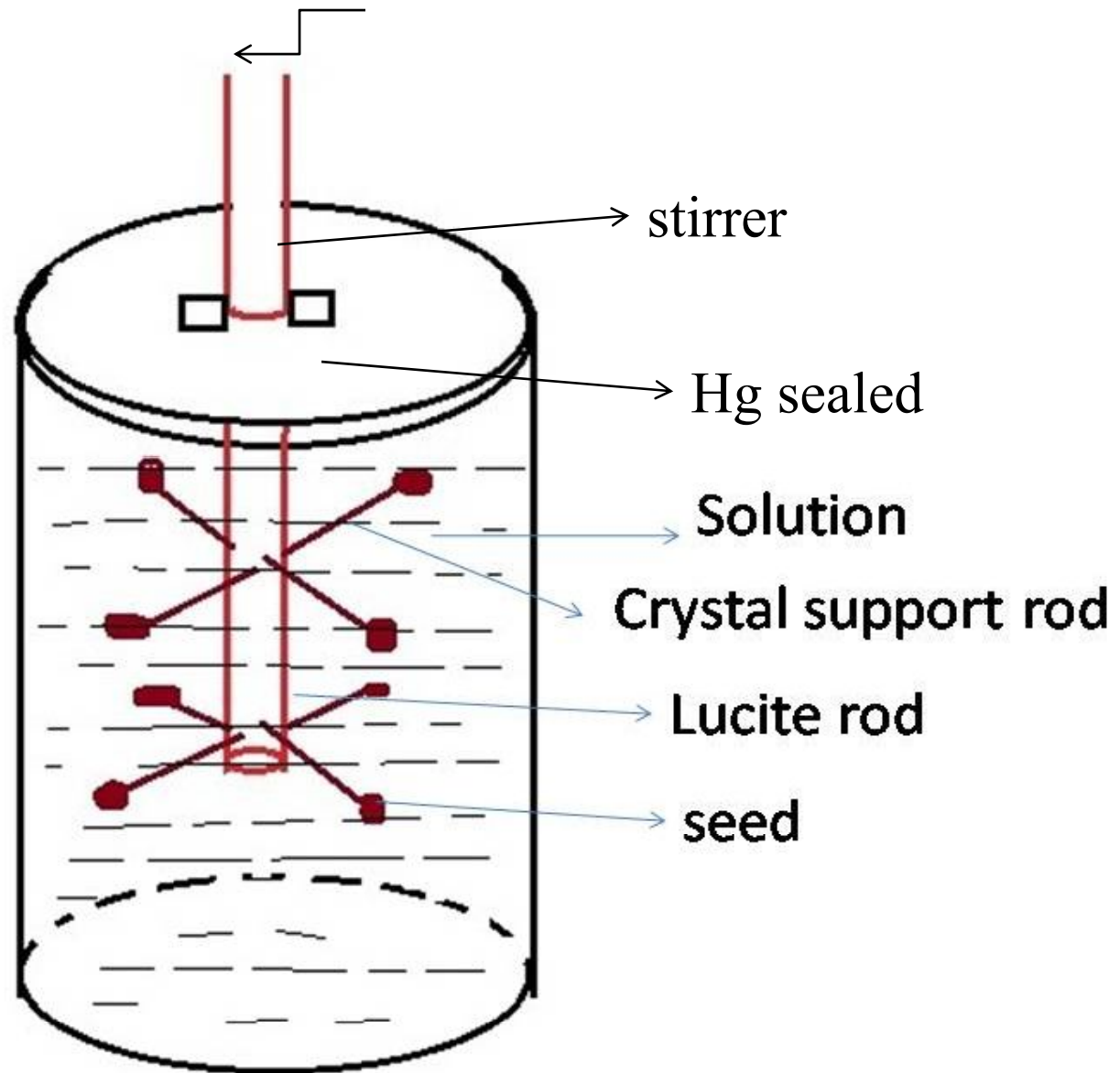


# HOLDERS ROTARY METHOD

(slow cooling method)

First step : preparation of saturated solution

# HOLDERS ROTARY METHOD



- Most useful method to grow crystals by slow cooling technique
- Saturated or near saturated solution added to tank T1
- Suitably oriented seeds are mounted on the seed holder
- Some cases crystals will form on the wall ,To avoid this stirrer the rotary crystalliser during cooling

- Saturation of the solution can be confirmed by inserting a tungsten rod into the solution
- If the current is descending, the solution is denser than the surrounding fluid
- If the current is rising, the fluid is more dense

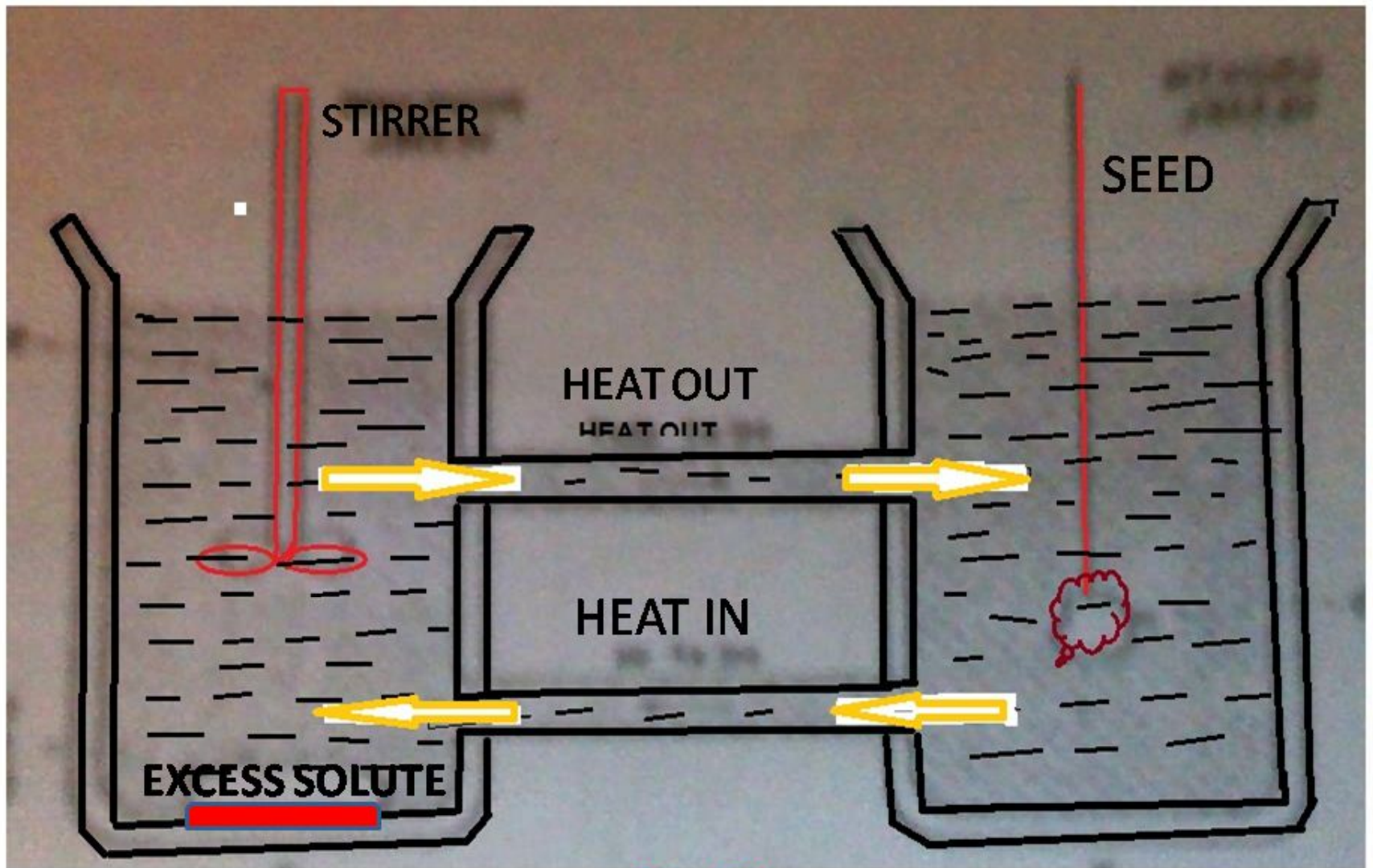


- Temperature is maintained at the desired level by heater controller arrangement, i.e., mercury thermoregulator
- Cooling rate 0.1-1.5 degree
- The cover is sealed at the top to prevent evaporation

# TEMPERATURE DIFFERENTIAL METHOD

(slow cooling method)

---KRUGER AND FINKE



T 1  
DISSOLVING VESSEL

$$T1 > T1$$

T 2  
GROWTH VESSEL

This method involves the transport of the materials from the hot region containing the source material to be grown, to a cooler region where the solution is supersaturated and the crystal grows

First step : preparation of saturated solution

- Bring the growth and dissolving vessels to the same temperature
- And fill the saturated solution in it
- Test the solution in the growth vessel whether it is saturated or not

Then raise the temperature in the dissolving vessel slightly  $T_2 < T_1$

Now insert the seed crystal in growth vessel

Maintain growth vessel cooler than dissolving vessel  
For the purpose of slow cooling process

Crystal will start to grow in seed as the process of nucleation

The solution will be under saturated and the surface damage on the seed ,attached nucli ,floating nucli etc will be form

Thus nucleation will be form on the seed

The tube connecting the vessels serve heat exchangers



Used to piezoelectric applications such as growth of Ammonium dihydrogen phosphate and Ethylen diamine tartrate