

Exp. No. 2

Crystallization & recrystallization

Crystallization is the process of precipitating crystals from a solution due to changes in solubility conditions of the solute in the solution. This is a separation technique which is similar to regular precipitation.

Precipitates are solids consisting of particles in a solution. Sometimes solids are a result of a chemical reaction in a solution. These solid particles will eventually settle down due to their density, and it is known as a precipitate. In centrifugation, the resulting precipitate is also known as the pellet. The solution above the precipitate is known as the supernatant. The particle size in the precipitate changes from occasion to occasion. Crystals can be easily filtered, and they are larger in size.

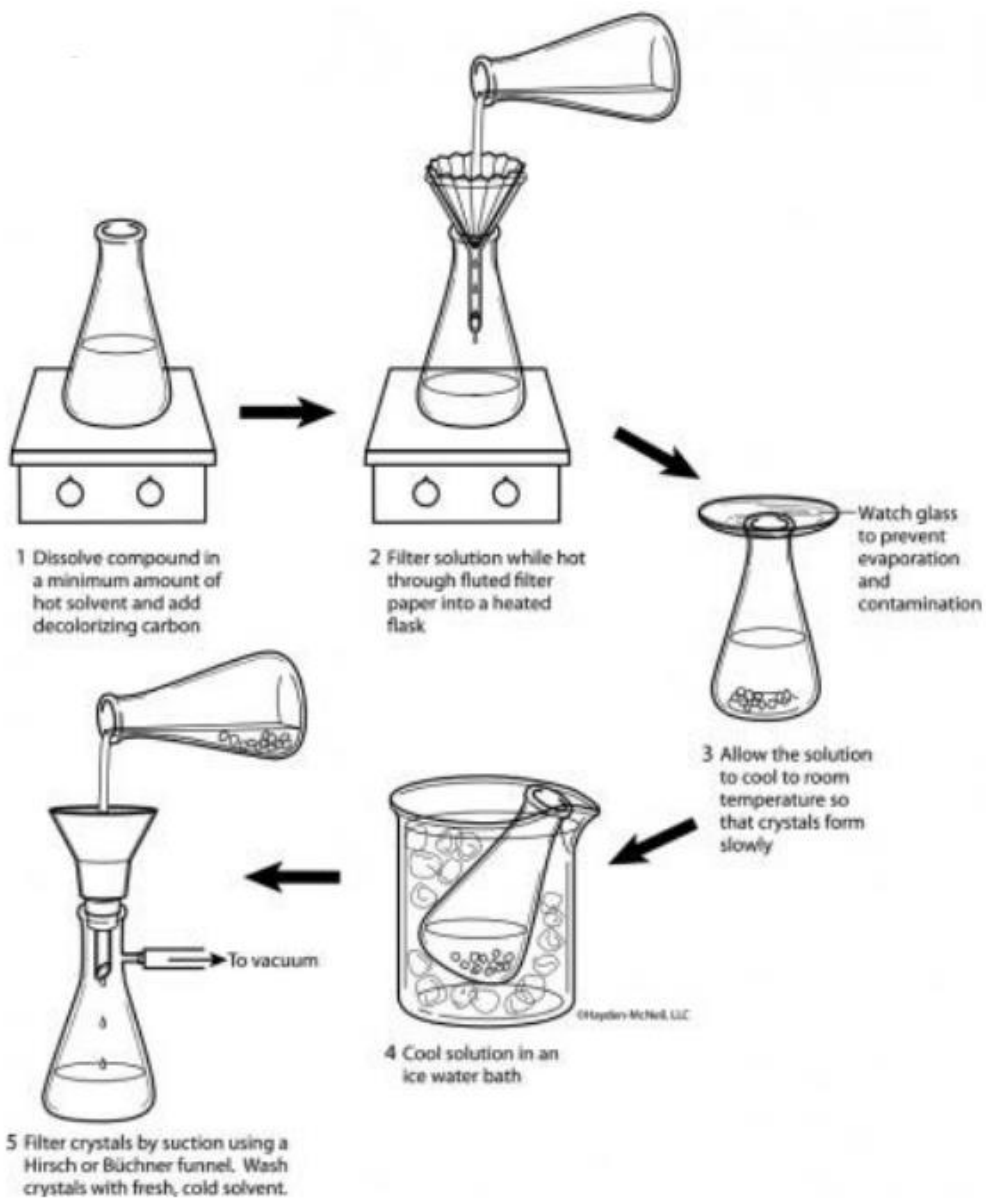
The difference in crystallization method from the normal precipitation is that, the resulted solid is a crystal. Crystalline precipitates are more easily filtered and purified. The crystal particle size can be improved by using dilute solutions and adding the precipitating reagent slowly while mixing. The quality of the crystal and the improvement in filterability can be obtained from the dissolution and recrystallization of the solid. Crystallization can be seen in nature too. It is most often carried out artificially for various types of crystal production and purification.

Recrystallization is a technique to purify the crystals obtained from crystallization method. Though crystallization separates the compound in almost pure form, when the crystals form some of the impurities may trap in it. By recrystallization method, these impurities can be removed to a greater extend.

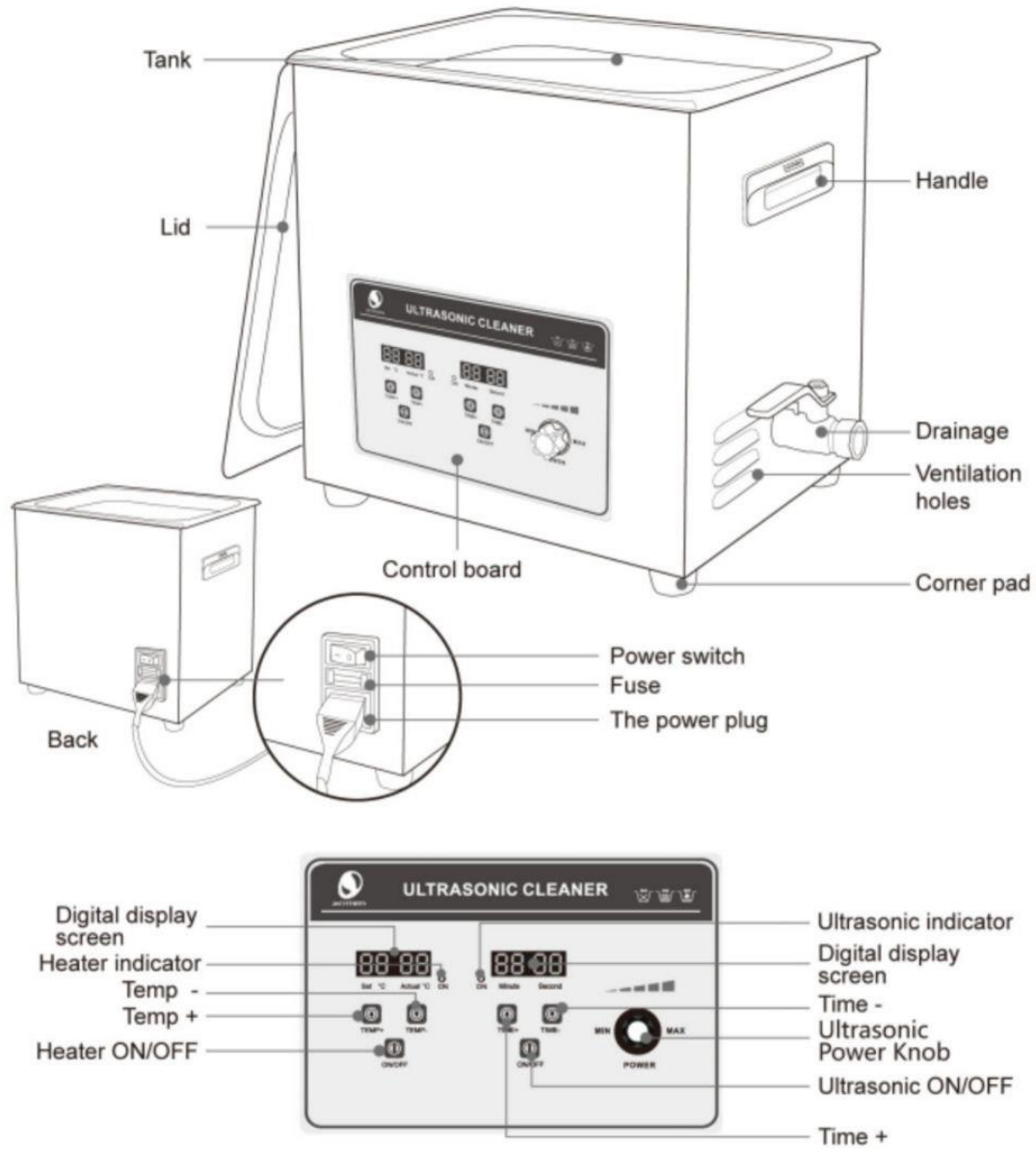
Normally the crystals are dissolved in very little amount of hot solvent and allowed to dissolve completely. When this is allowed to cool slowly (may be after filtering), the crystals may appear again. These crystals are free of the impurity. The crystals can be separated by filtering the solution again. Recrystallization process can be carried out in several ways, and several times to increase the purity of the desired crystal.

Procedure:-

- 1. Weigh 5 grams of impure substance in a small beaker .**
- 2. Dissolve in a minimum amount of hot solvent .**
- 3. Filter while the solution is hot through short neck funnel, using fluted filter paper .**
- 4. Cool the solution by using skymen ultrasonic cleaner substituted cooling bath until the crystals will appear .**
- 5. Filter off the crystals through Buchner funnel .**
- 6. Recrystallize again as above (step 2 – 5) , and finally keep to dry .**
- 7. Check the melting point and determine the percentage of yield .**



POWER ADJUSTABLE MODEL



Skymen ultrasonic cleaner

OPERATION STEPS

◆ Before Operating Your Cleaner



1. add cleaning liquid

Fill the tank with warm or tap water below the maxline; Add required quantity of cleaning solution. When using flammable liquid such as alcohol, gasoline and so on, heating is strictly forbidden! ultrasonic vibration for a long time cause temperature of liquid increase, caution fire!



2. open power

Plug the cleaner into a grounded outlet. Turn on the power switch at the tank back, then machine is power on. ("O" is closed, and "I" is open)

◆ Start ultrasonic cleaning



MECHANICAL MODEL

Start(ULTRASONIC), clockwise rotate to choose the time you required between 0~30 min. When the indicator light is on and make "ZIZI" sound, it shows ultrasonic operation work OK.



POWER ADJUSTABLE MODEL

the time is random display for primary starting. Touch TIME+ once to increase time by 1 minute; Touch TIME- once to decrease time by 1 minute. Touch ON/OFF switch after set the time, ultrasonic indicator turns red and machine starts working. Timer will count down and stop work when time shows "00:00". Timer display shows the previous setting time when next operation

◆ Heat



MECHANICAL MODEL

If need heating, start(HEATING) to adjust required temperature, normally 40~60°C is the best. (Heating is optional upon needs)

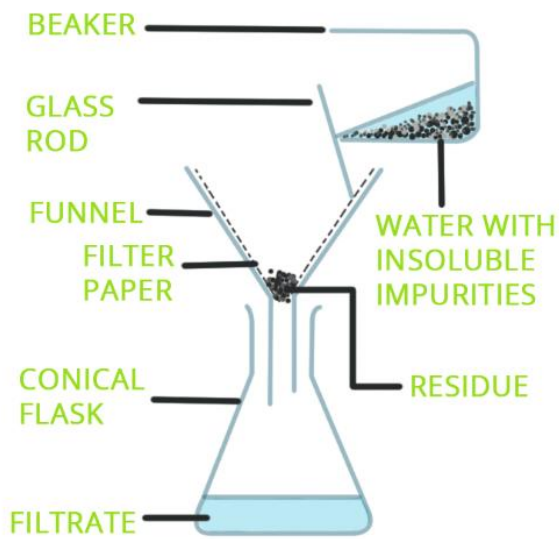


POWER ADJUSTABLE MODEL

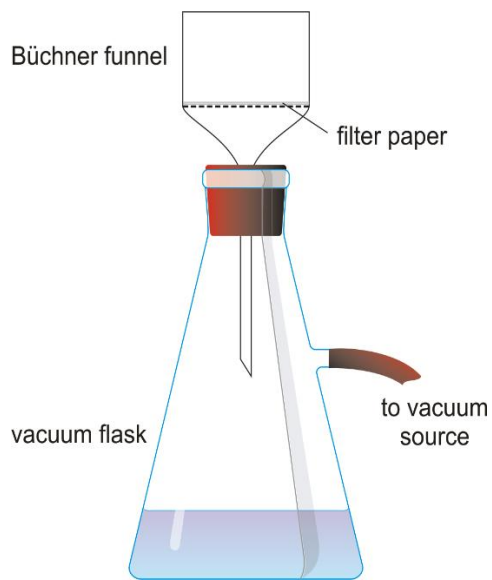
The setting temperature is random display for primary starting and Actual is rom temp. Touch TEMP+ once to increase temperature by 1°C; Touch TEMP- once to decrease temperature by 1°C. Temperature display shows setting temperature and actual temperature when heater is working, the heater indicator goes off when actual temperature reaches setting temperature.

◆ Adjusting power

The function is only available for power adjustable models! Rotate SLOWLY the power knob clock-wise to increase the power from 40% to 100%, and counter-clockwise SLOWLY is to decrease sonic power.

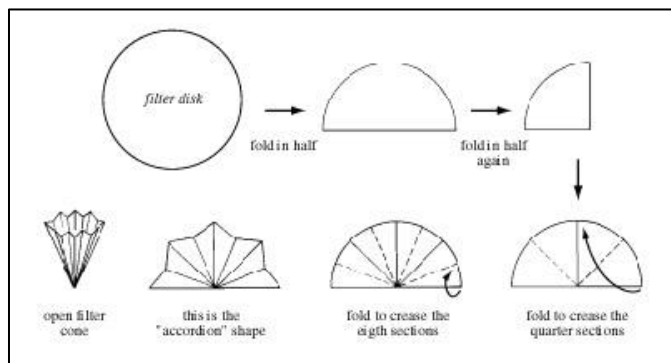


Slow filtration



Buchner flask and funnel

Fast filtration



Fluting a filter paper

Discussion:

- 1-comparison of slow filtration and fast filtration with drawing.**
- 2- comparison of crystallization and precipitation.**
- 3- The benefit of using fluted filter paper.**
- 4- descriptions of suitable solvents used in recrystallization.**
- 5-scratching and seed method meaning .**
- 6-whats benefit of Skymen ultrasonic cleaner device and using it?**

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