

Aim:- To perform the crystallization by cooling.

Reference :-

Requirements :-

- Test tubes (10) KNO_3
- Test tube stand (1) ICE
- Hot water bath (2)
- Thermometer (2)
- Beaker (250 ml) (1)
- Stirrer (1)
- Tripod stand (1)
- Funnels (3)
- Filter papers
- Weighing balance.

Theory :-

Crystallization is a process of spontaneous arrangement of particle into repeatedly arrange geometrical pattern from a supersaturated solⁿ. Crystallization can occurred if solⁿ is in a supersaturated state. The supersaturation can be produced by either of these following ways.

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S.No.	Time (min)	Weight of empty filter paper (W_1)	Weight of filter paper with crystals (W_2)	Weight of crystals $W_3 (W_2 - W_1)$	% Weight of crystals
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					

- (i) Super saturation by cooling.
- (ii) Super saturation by the evaporation of the solvent.
- (iii) Super saturation by adiabatic evaporative cooling.
- (iv) Super saturation by adding a substance that reduces the solubility of the substance in the solⁿ.

This experiment is based on crystallization by cooling and is only applicable to the solute whose solubility increases with increase in ~~temp~~ temp. cooling can be done by 2 methods.

- (i) Shock cooling.
- (ii) Slow cooling.

Rate of crystallization during shock cooling is fast and the size of the crystals produced is small. But during slow cooling the rate of crystallization is slow and it will achieve comparatively big crystals. Saturated solⁿ are prepared by elevating the temp. ~~temp~~ temp of the solⁿ. During cooling the solubility of the solute decreases as a result the dissolved solute starts to crystallize. The yield is dependent on the time of contact and the temp. yield is calculated in terms of %age. A graph can also be plotted by taking

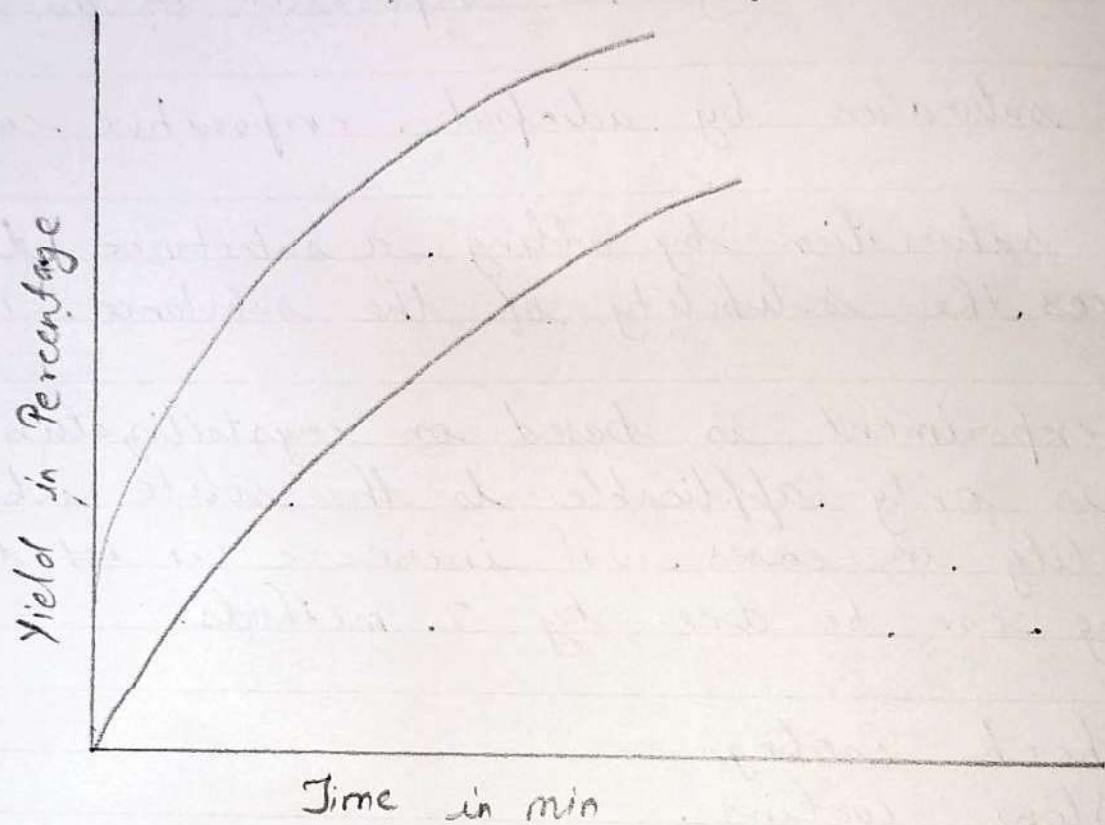


Fig :- Model graph showing comparisons of yield obtained by shock cooling and slow cooling with time.

time on x-axis and yield in percentage on y-axis.

Procedure:

- i) Weigh 150 gm of KNO_3
- ii) Take 100 ml of water in beaker.
- iii) Keep the beaker in a water bath at 60°C .
- iv) Slowly add the KNO_3 in small portions in to the beaker containing water.
- v) Stir the solⁿ continuously and stop the addition of solute when a small portion of the solute remains undissolved at the bottom.
- vi) Weigh the remaining quantity of the KNO_3 and find out the amount of KNO_3 used to produce the saturated solⁿ.
- vii) Note down the amount of KNO_3 used to produce a saturated solⁿ at elevated temp^t. (i.e. x gm in 100ml)
- viii) Keep the solⁿ in the same water bath for five min to ensure the saturation.
- ix) Mark and Arrange 10 test tubes in a test tube rack

- x) Add 10 ml of hot ~~st~~ saturated in to all of the test tubes.
- xi) Keep the entire test tube immediately into an ice bath.
- xii) Take out the first test tube after the commencement of 10 min.
- xiii) Filter the solⁿ present in the test tube (using pre weighed filter paper).
- xiv) The ~~graphs~~ crystals in the filter paper are kept for air drying. (a hair dryer can also be used for drying the crystals.)
- xv) After drying the crystals find out the yield.
- xvi) Find out the percentage yield (Amount of crystals produced from 10 ml amount of solute present in 10 ml) $\times 100$
- xvii) Take out - 2nd, 3rd, 10th test tubes at an interval of 15, 20, 25, 30, 60 min respectively.
- xviii) The same procedure - 13-16 is repeated for all the solⁿ.

- xix) A graph is plotted by taking percentage yield at different time interval on Y-axis and time in min on X-axis.
- xx) Repeat the steps 1-10.
- xxi) Now keep the test tubes in the normal atmosphere temp.
- xxii) After 10 min filter the content present in the first test tube.
- xxiii) Repeat the steps 14-16.
- xxiv) Take out 2nd, 3rd, 4th 10th test tubes at an interval of 15, 20, 25, 30 min respectively.
- xxv) Repeat the steps 13-16.
- xxvi) A graph is plotted by taking percentage yield at different time interval on Y-axis and time in min on X axis.
- xxvii) Compare the two graphs.

Result :-

The % of KNO_3 crystals by shock cooling after 45 min =

The percentage of KNO_3 crystals by slow cooling after 45 min =

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