PROJECT

- SUB PHYSICAL CHEMISTRY
- B .SC CHEM(H) 2 ND YEAR
- ROLL NO 127

PREPARED BY

SWATI JHA

AIM

TO DRAW THE FACE DIAGRAM OF B NAPHTHOL RESORCIONAL SYSTEM BY MELTING POINT METHOD

APPARATUS

CAPILLARY TUBE, MELTING POINT APPARATUS,WATCH GLASS,SPATULA ,TEST TUBES,WEIGHING APPARATUS

PURPOSE OF USING ALL THESE APPARATUS

CAPILLARYTUBE

SINCE WE ARE USING MELTING POINT APPARATUS, CAPPILARY TUBE CAN ONLY BE USED FOR THIS PURPOSE BECAUSE SIZE OF THE HOLE PRESENT ON THE MELTING POINT APPARATUS IS EQUIVALENT TO THE THICKNESS OF THE CAPPILARY TUBE.

MELTING POINT APPARATUS

WE ARE USING MELTING POINT APARATUS BECAUSE IT IS MORE CONVINIENT,ACCURATE ,SAFER THAN THE TRADITIONAL METHOD USING ZEHLAD FLASK FILLED WITH CONC SULPHURIC ACID TO WHICH CONSTANT HEATING WITH FLAME WAS REQUIRED WHICH IS VERY RISKY.

TEST TUBES

TEST TUBES WERE USED TO MELT THE REQUIRED COPOSITION OF MIXTURE, SO THAT TWO CONSTITUENTS(B-NAPHTHOL AND RESORCINAL) CAN MIX PROPERALY WITH EACH OTHER

WATCH GLASS AND SPATULA

TO CRUSH THE MELT INTO FINE POWDERER SO THAT IT CAN BE FILLED IN CAPPILARY TUBE

WHY WE ARE USING MELTING POINT METHOD?

- We have used only .5 g of powdered chemicals ,which is more than sufficient to get melting point of composition using capillary tube.

- To verify the fact that the temp at which melting of composition starts follows the same trend as the solidification of the melt

Theory: FOR THE SOLID SOLUTION OF SOLUTES IN SOLVENT.

solid solution is a solid-state solution of one or more solutes in a solvent. Such a mixture is considered a solution rather than a compound when the crystal structure of the solvent remains unchanged by addition of the solutes, and the mixture remains in a single homogeneous phase.

Types of solid solution curve

1. Minimum type of solid solution
2. maximum type of solid solution
3. No maximum or minimum type of solid solution

Types of incorporation in solid solution: FOR SOLUTES IN SOLVENT

1. Substitution solid solution

   The solute may incorporate into the solvent crystal lattice by replacing a solvent particle in the lattice.

2. Interstitial solid solution

   The solute molecule may incorporate by fitting into the space between the solvent particle.

- Both of these types of solid solution affect the properties of the material by distorting the crystal lattice and disrupting the physical and electrical homogeneity of the solvent material.

Property of substances to form solid solution.

- Some mixtures will readily form solid solutions over a range of concentrations, while other mixtures will not form solid solutions at all. The propensity for any two substances to form a solid solution is a complicated matter involving the chemical, crystallography, and quantum properties of the substances.

Solid solution (defination)

A homogeneous crystalline structure in which one or more types of atoms or molecules may be partly substituted in the original atoms and molecules without changing the structure.

Types of solid solution

1. *Substitutional solid solution*: chemical variation is achieved simply by substituting one type of atom in the structure by another.

2. *Coupled substitution*: this is similar to the substitution solid solution, but in a compound Cations of different valancy are interchanged. To maintain charge balance, two coupled cation substitutions must take place.

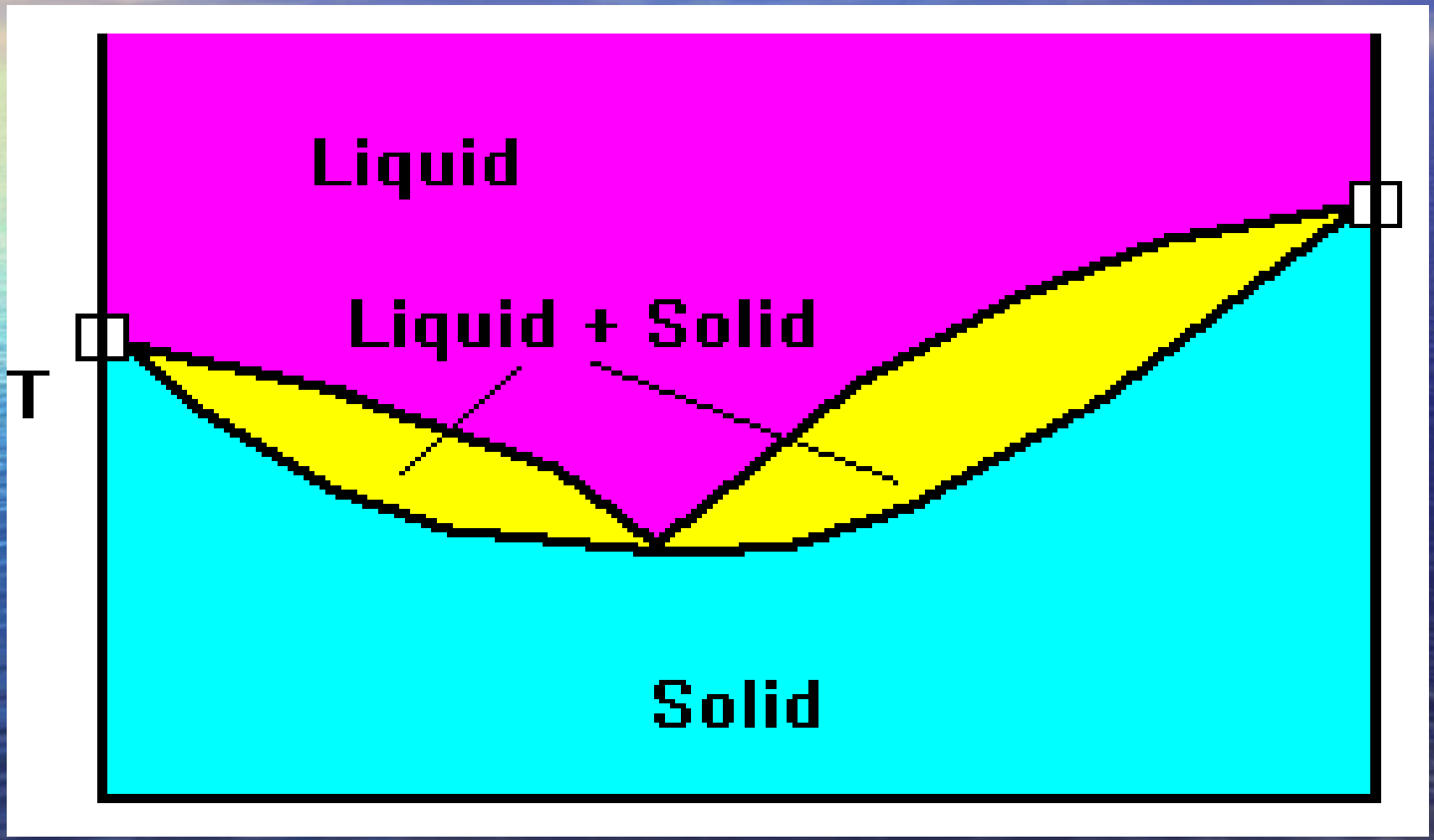
3 .*Omission solid solution*: chemical variation is achieved by omitting cations from cation sites that are normally occupied.

4. *Interstitial solid solution*: chemical variation is achieved by adding atoms or ions to sites in the structure that are not normally occupied.

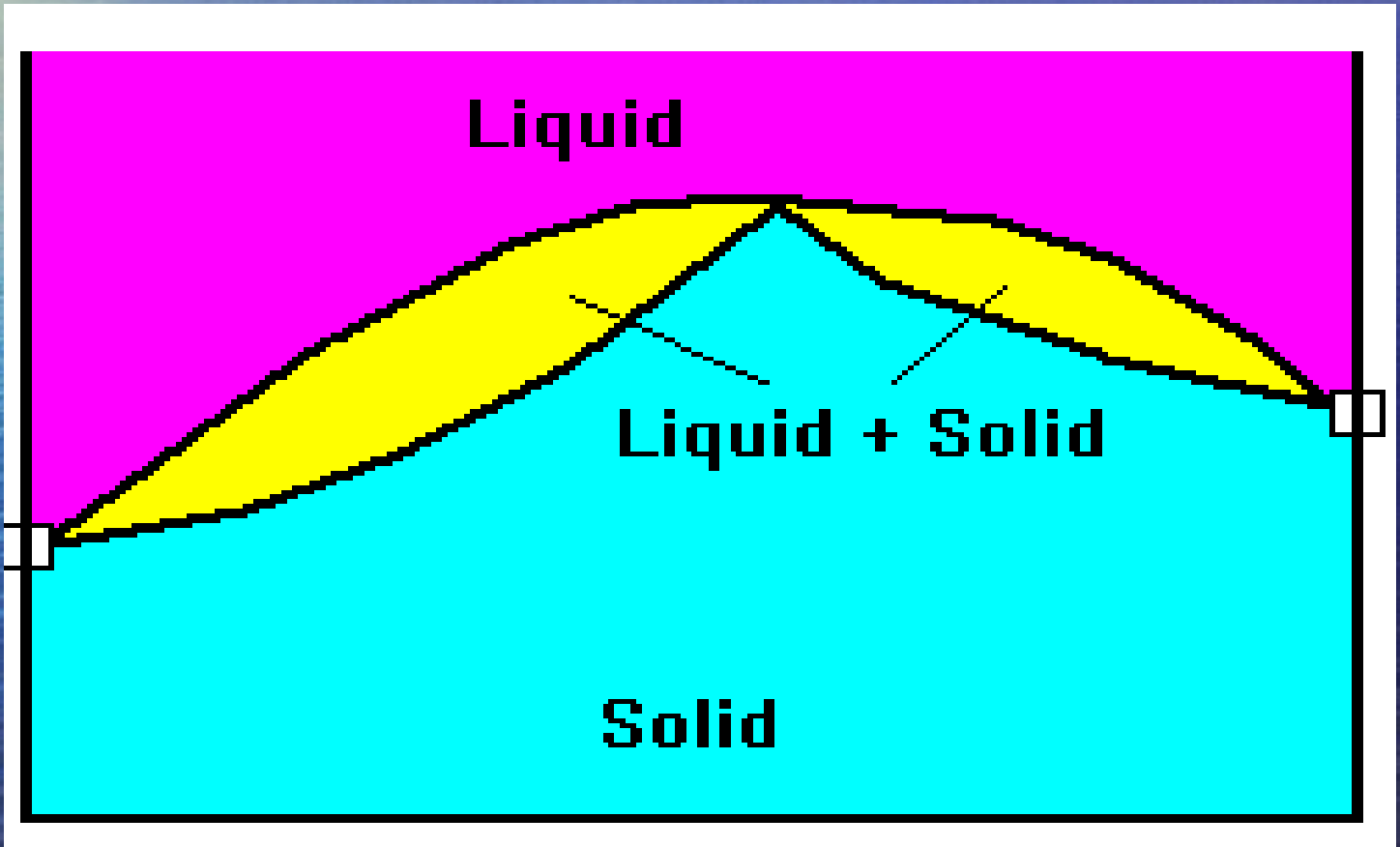
Maximum and minimum type solid solution

- Sometimes solid solutions can have maxima or minima between the end members. These are no problem: the solid and liquid compositions will still slide down the liquidus and solidus curves. Treat each section as a simple solid solution.

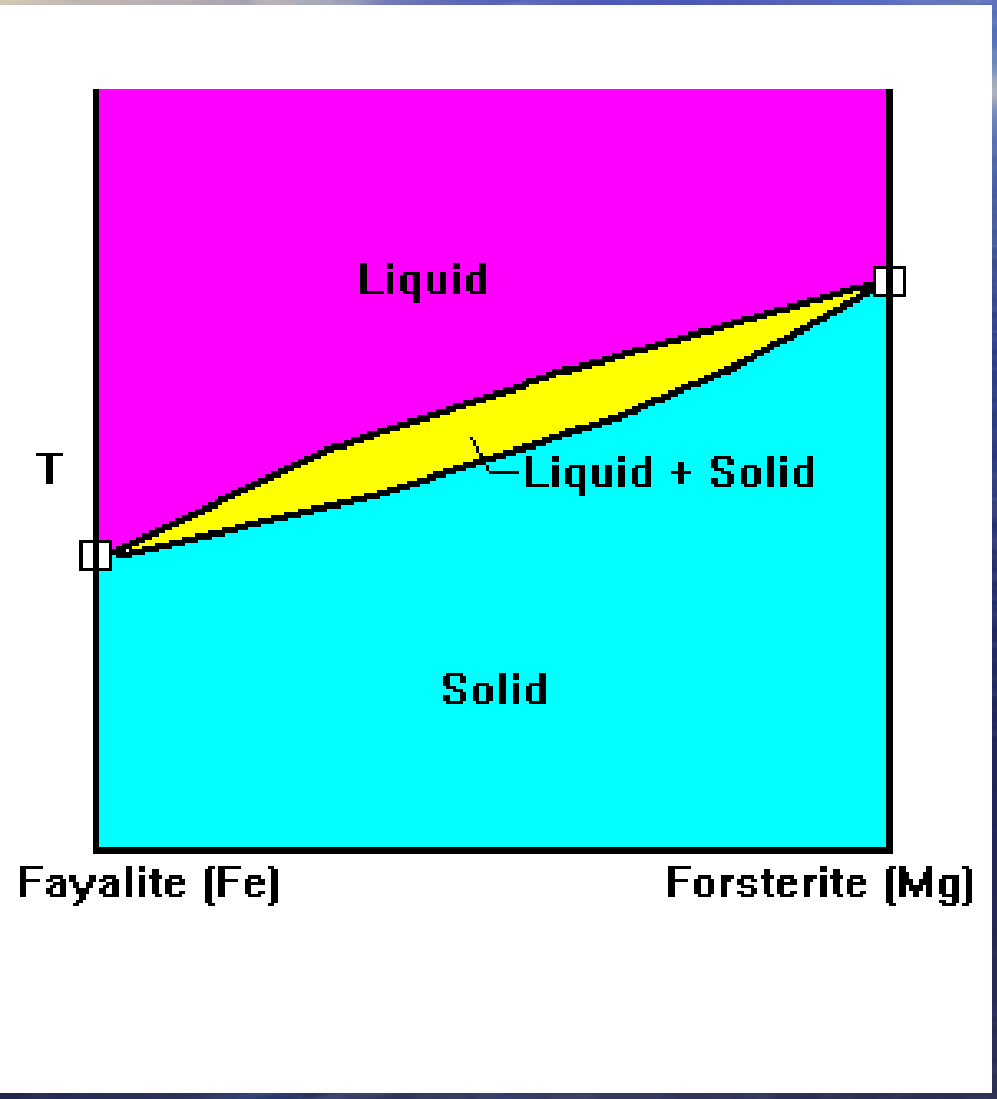
Minimum Type Solid Solution



Maximum type Solid Solution



Solid Solution With No Maximum or Minimum.



- The system plots in the purple field it is all liquid, in the yellow field it is a mixture of liquid and solid, and in the blue field it is entirely solid. The upper bound of the liquid+solid field is called the *liquidus*, and the lower bound is called the *solidus*.
- In a simple binary eutectic, if you know the melting points of the end members and the position of the eutectic, you can largely predict the system, though the exact curvatures of the liquidus curves have to be determined experimentally. With a solid solution.

Factors affecting solid solution state

1. *Atomic/ionic size*: If the atoms or ions in a solid solution have similar ionic radii, then the solid solution is often very extensive or complete. Generally, if the size difference .is less than about 15%, then extensive solid solution is possible. For example, Mg^{2+} and Fe^{2+} have a size mismatch of only about 7%, and complete solid solution between these two elements is observed in a wide range of minerals. However, there is a 32% size difference between Ca^{2+} and Mg^{2+}, and we expect very little substitution of Mg for Ca to occur in minerals.

2. Temperature: High temperatures favour the formation of solid solutions, so that endmembers which are immiscible at low temperature may form complete or more extensive solid solutions with each other at high temperature. High temperatures promote greater atomic vibration and open structures, which are easier to distort locally to accommodate differently-sized cations. Most importantly, solid solutions have a higher entropy than the endmembers, due to the increased disorder associated with the randomly distributed cations, and at high temperatures, the -*TS* term in the Gibb's free energy stabilises the solid solution

3. *Structural flexibility*: Although cation size is a useful indicator of the extent of solid solution between two endmembers, much depends on the ability of the rest of the structure to bend bonds (rather than stretch or compress them) to accommodate local strains.

4. Cation charge : Heterovalent substitutions (i.e. those involving cations with different charges) rarely lead to complete solid solutions at low temperatures, since they undergo complex cation ordering phase transitions and/or phase separation at intermediate compositions. These processes are driven by the need to maintain local charge balance in the solid solution as well as to accommodate local strain

procedure

1. the different composition mixture of B-naphthol and recorcinal were made according to table (1).
2. Cappilary tube was packed with small quantity of recorcinol.
3. the filled cappilary was then kept in the melting point apparatus

4.Noted the temperature at which melting had started and finished.

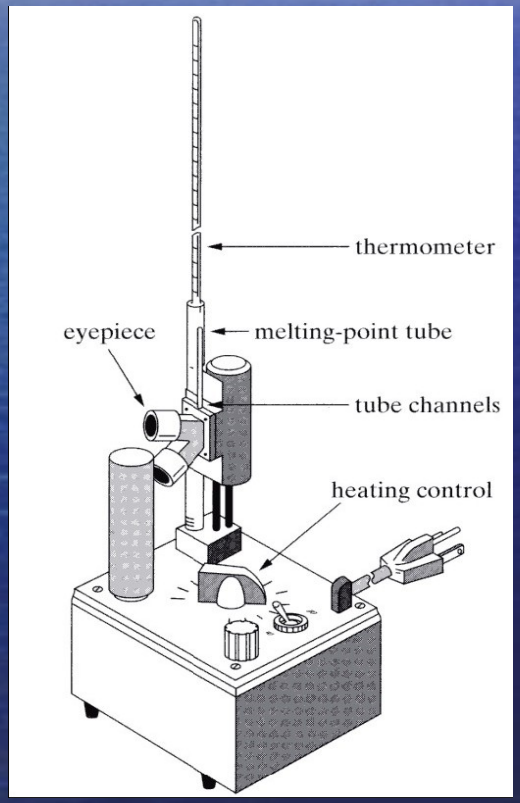
5.repeat the same procedure for all the composition made according to table(1).

6. plotted a graph between temperature on y-axis and composition on x-axis.

Packing a capillary for melting point determination

Thin-walled capillary melting point tubes are used to hold melting point samples. This tube needs to be sealed at one end (pre-sealed tubes should be available in the laboratory, or, an open capillary can be sealed by inserting the tip into a Bunsen flame near the base of the flame). To pack the tube, the open end is pressed gently into a small amount of the sample of the crystalline material on a watch glass or weighing paper. To transfer the crystals from the open end to the bottom of the tube, tap the bottom gently on the bench top or scratch the top edge of the tube on a small file or a coin with a milled edge. A densely packed column of crystals about 3 mm high in the tube is all that is required. A packed capillary attached to a normal mercury thermometer is shown to the left.

Melting Point Apparatus



Obeservation table(1)

Composition(%)	B-naphthol(%)	Recorcinol (%)	B-naphthol(g)	Recorcinol(g)
0	0	100	0	.5
10	10	90	.05	.45
20	20	80	.1	.4
40	40	60	.2	.3
60	50	50	.25	.25
70	60	40	.3	.2
80	70	30	.4	.1
Composition(%)	B-naphthol(%)	Recorcinol (%)	B-naphthol(g)	Recorcinol(g)

Obeservation table(1)

A CTUAL MELTING POINT OF RESORCINOL IS (109-111) DEG CELCIUS AND (121-123) DEG CELCIUS FOR B-NAPHTHOL.

TABLE FOR THE FORMATION OF DIFFERENT COMPOSITIONS

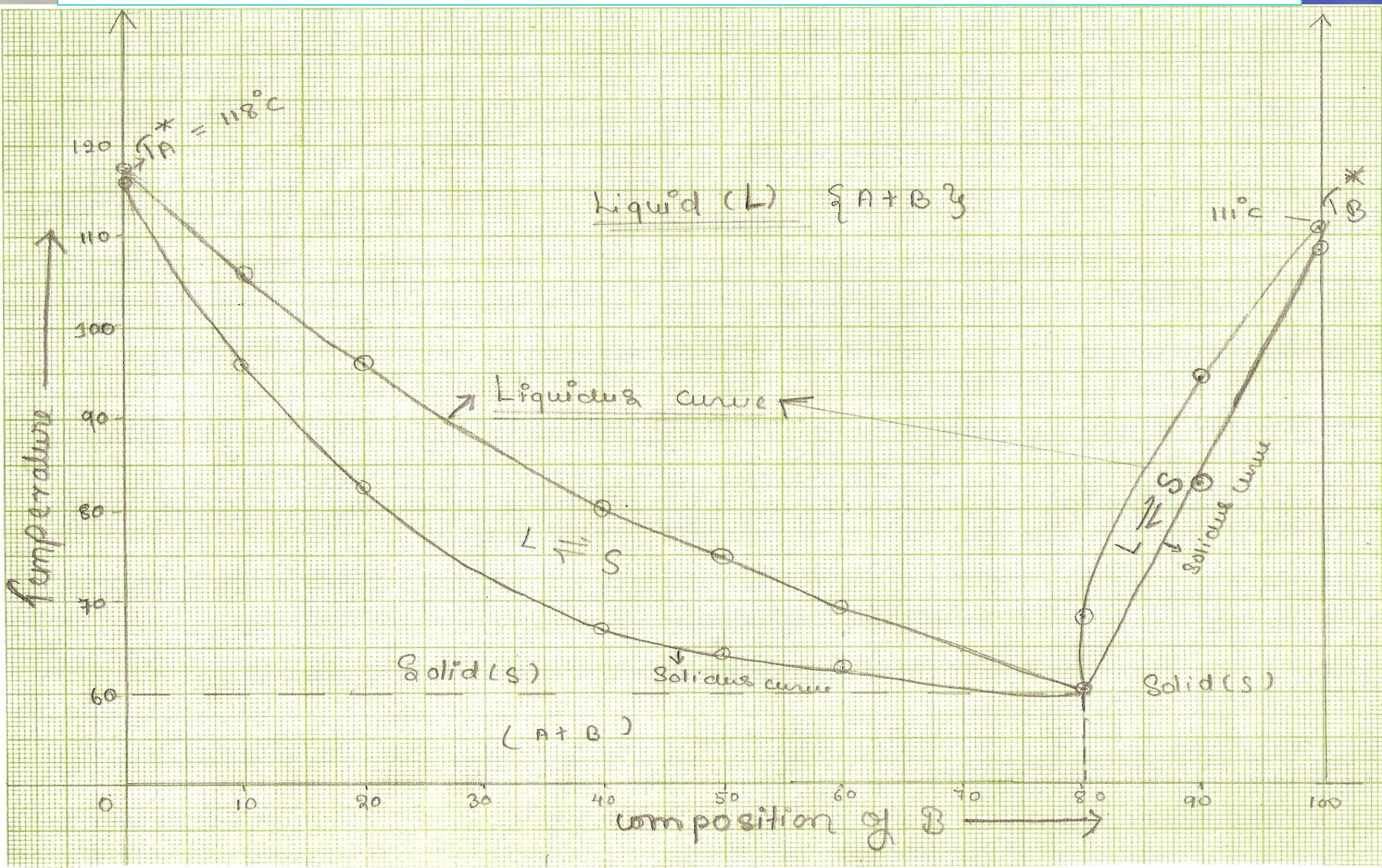
Mass% of B	B-naphthol(%)	Recorcinol (%)	B-naphthol(g)	Recorcinol(g)
0	0	100	0	.5
10	10	90	.05	.45
20	20	80	.1	.4
40	40	60	.2	.3
60	50	50	.25	.25
70	60	40	.3	.2
80	70	30	.4	.1
Mass% of B	B-naphthol(%)	Recorcinol (%)	B-naphthol(g)	Recorcinol(g)

OBSERVATION TABLE(2):FOR THE DETERMINATION OF MELTING POINTS

Mass%of B	Compositio n a+b (g)	Melting Point(start)	Melting point(end)
0	.5A	116	118
10	.45A+.5B	96	106
20	.4A+.1B	85	96
40	.3A+.2B	67	80
50	.25+.25	64	75
60	.2B+.3A	63	69
80	.1A+.4B	60	76
Mass%of B	Compositio	Melting	Melting

GRAPH: PLOT OF TEMPERATURE VS COMPOSITION S

Y-AXIS 1 SMALL DIVISION=.5 DEGREE CELCIUS , X-AXIS 1 SMALL DIVISION=.5 MASS%



Precautions:

1. the ingradient should pack properly in the capillary tube.

2.do not keep the capillary tube when the temperature of the melting point apparatus is already at very high temperature.

3.do not take much amount in the capillary tube.

It will give range or incorrect melting point.

4.Prevent your ingradient to be mixed with any impurities.

RESULT

- MINIMUM TYPE SOLID SOLUTION WAS OBTAINED WITH B-NAPHTHOL AND RESORCINOL SYSTEM.
- MELTING POINT OF PURE RESORCINOL IS FOUND TO BE (109-111) DEGREE CELCIUS
- MELTING POINT OF B-NAPHTHOL IS FOUND TO BE (116-118) DEGREE CELCIUS
- MINIMUM FREEZING POINT (INVARIENT POINT) IS FOUND TO BE 60 DEGREE CELCIUS
- COMPOSITION OF INVARIENT POINT IS 80% OF RESORCINOL AND 20% OF B -NAPHTHOL

CONCLUSION

- I THINK I HAVE GOT FINE RESULT.
- THE INVARIENT POINT WHICH I HAVE OBTAINED IS LESS THAN THE MELTING POINT OF THE INGRADIENTS USED (B-NAPHTHOL AND RESORCINOL). WHICH IS THE MAIN CHARACTERISTIC OF MINIMUM TYPE SOLID SOLUTION.
- THE MELTING POINT OF CRUDE OBTAINED IS ALMOST EQUAL TO THE ACTUAL MELTING POINT OF THE INGRADIENTS.
- CONTINIOUS SMOOTH CURVE IS OBTAINED.