

PHASE DIAGRAMS IN VIVO

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INTRODUCTION

Fluid Inclusions

All solids crystallizing from a liquid phase trap small, accidental amounts of the parent liquid in features we call fluid inclusions (Fig. 1). Fluid inclusions typically range in size from less than 1 micron to several hundred microns and are easily seen in transparent minerals with a standard high-power petrographic microscope (200 to 400X, condenser in). Excellent fluid inclusions can be found in almost any sample of fluorite (cleave off a fragment no more than 2 mm thick) and quartz.

Fluid inclusions are time capsules that preserve small volumes of the liquid from which the mineral precipitated. By studying the contents of a fluid inclusion using simple heating / freezing experiments the temperature and pressure at which the mineral formed can be determined. The density and composition of the fluid can also be determined directly! In recent years it has become possible to extract the fluid from single inclusions and determine its isotopic composition and in some cases its age. Fluid inclusion studies, therefore, provide a wealth of data and are used to decipher the P-T-t- ρ history of mineral and rocks.

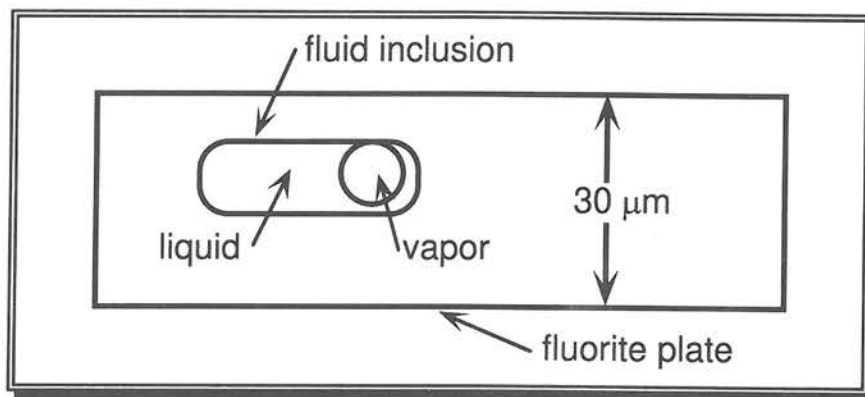


Figure 1. Idealized photomicrograph of fluid inclusions in fluorite at room temperature. Natural inclusions containing a low salinity fluid trapped above room temperature typically contain two phases-- aqueous fluid and vapor bubble. The bubble is actually a “shrinkage” phenomenon. Assume an aqueous fluid was trapped at 250°C. The fluid would have a density of 0.8 gm/cm³. As the temperature drops both the host fluorite and the aqueous fluid will contract (thermal expansion). It is well known that solids have the least thermal expansion and that gases have the greatest thermal expansion. Liquids are intermediate but closer to solids. As the temperature reaches room temperature the density of the liquid increases to 1.0 gm/cm³ (assume that nothing enters or leaves the fluid inclusion). Over the temperature range considered the density and volume change of the fluorite is negligible. Since the mass of fluid remains constant while the density increases it follows that the volume must decrease (volume = mass / density). The fluid separates into a vapor and fluid phase such that the total mass remains the same at room temperature ($vol_{vapor} * density_{vapor} + vol_{fluid} * density_{fluid}$) as at the entrapment temperature ($vol_{fluid} * density_{fluid}$). What must the volume of the bubble be if the density of the vapor is 0.01 gm/cm³?

The significance of fluid inclusions was recognized early on but quantitative results from fluid inclusion studies awaited the completion of laboratory experiments in geologically relevant systems. In an interesting turn of events many laboratory experiments are now carried out using synthetic fluid inclusion as the experimental vessels. The advantage of using synthetic fluid inclusions rather than steel pressure vessels is that the contents can be examined visually at all times, equilibrium is established faster (lower mass), safety is greater (lower volume), and cost is lower.

Phase Diagrams for Brines, Magmas, and Minerals

Phase diagrams summarize experimental and/or observational data on mineral associations and mineral (and fluid) compositions. These data are used to deduce mineral reactions and to quantify geochemical variables in mineral reactions (T , pH , P , bulk composition, X_{mineral} , X_{fluid} , etc.). There are many types of phase diagrams (pressure - temperature, temperature - pH , activity - activity, temperature - activity, etc.) and they are all used to learn about various Earth processes.

Temperature-Composition Diagrams. This laboratory exercise explores temperature-composition (T - X) phase diagrams. Figure 2a shows a typical phase diagram that describes the melting relations in the **chemical system** A - B. Temperature is shown on the vertical axis and composition of all phases are indicated on the horizontal axis. The composition scale is in weight percent ranging from 100% A on the left to 0% A on the right (the scale could just as easily be reversed with % B increasing to the right). The composition of all solids that may exist in this chemical system can be described by reading the scale on the horizontal axis. This hypothetical chemical system, A - B contains three solids (phases) at low temperature: A, B and C. The composition of solids A, B and C are 100% A, 25% A and 0% A, respectively. T_{mA} and T_{mB} represent the **melting temperature** of each of compounds A and B, respectively. The melting point of a compound is a diagnostic characteristic such as specific gravity or hardness and can be used to unambiguously identify a compound. A mixture of the two compounds will begin to melt at the **eutectic** temperature, a temperature lower than the melting temperature of either compound alone. The eutectic temperature is a diagnostic characteristic of a chemical system (more than one compound). Given a eutectic temperature measurement it is possible to identify uniquely the chemical system (Table 1). For a fixed composition, such as **R** (Fig 2a), the temperature above which no solid remains is given by the **liquidus** (or saturation) curve. Above the saturation curve there will be no crystals because they all melt. At temperatures below the eutectic temperature only solid is present. Between the eutectic temperature and temperature given by the liquidus curve (for any specific composition) some material will be present as crystals and some as liquid (**2-phase field**). At a given temperature, such as T_1 it is possible to determine the proportions of solid and liquid (crystals and fluid). The horizontal line segment (**tie line** XZ at temperature, T_1) connects the composition of the solid on the left with the composition of the coexisting liquid on the liquidus curve. The tie line, XZ, can be divided into two shorter line segments XY and YZ. The proportion of liquid is given as the ratio of XY/XZ. The proportion of solid is given by the ratio of YZ/XZ. This is the **lever rule** (Brady and Stout, 1980). In the experiments described below the lever rule will be used to construct the liquidus curve from observed solid / liquid ratios.

Real T-X diagrams The phase diagram for ice-halite (H_2O - NaCl; Fig 2b) which is relevant to hydrologic applications closely resembles Fig 2a. Six phases are possible in this system -- The minerals ice, hydrohalite, and halite, and the liquids water (pure H_2O), NaCl liquid (not shown on diagram because it occurs above $40^\circ C$), and brine (H_2O - NaCl fluid). This phase diagram can tell us several important things. Here are some examples: (1) The melting point of ice is $0^\circ C$ exactly. (2) The north polar ice cap must be pure ice with no salt (except in fluid inclusions) in spite of forming from seawater (ice has no solid solution with NaCl)! (3) Once the temperature drops below $-20.8^\circ C$, it will do no good to salt the roads in order to melt the snow. (4) If a solution at room temperature contains salt crystals, then the salinity of the liquid must exceed 26.3 percent by

Figure 2a. Schematic temperature - composition (T-X) phase diagram. At temperature T_1 , the ratio of liquid to solid is given by the linear measures: XY/YZ . The fraction of the total that is liquid is $XY/(XY+YZ)$ or XY/XZ . Note that as temperature increases from T_1 line segment XY decreases in length indicating that the amount of solid decreases!

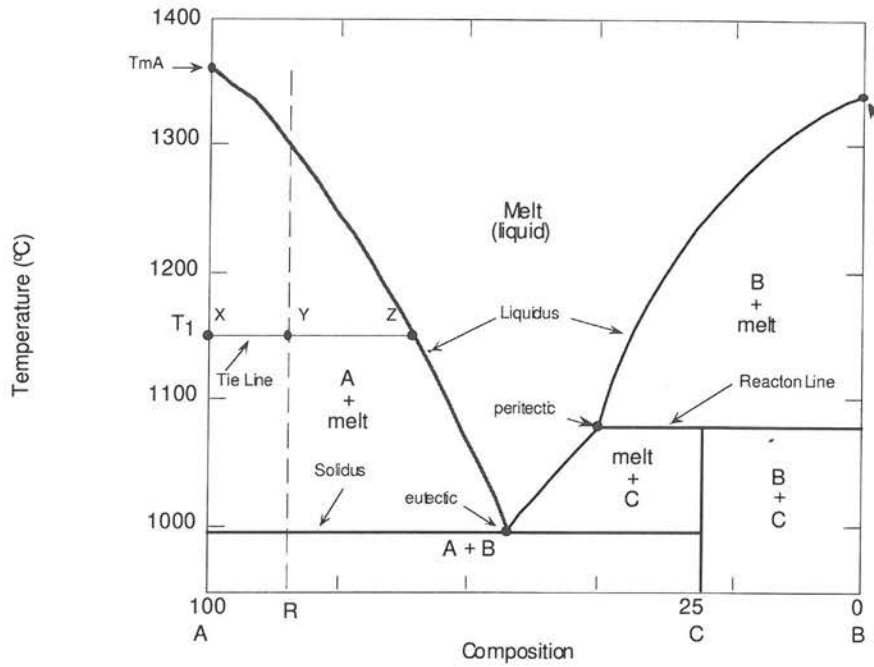


Figure 2b. The Ice - Halite System. The term liquid refers to brine (salt water). Modified from Crawford (1981).

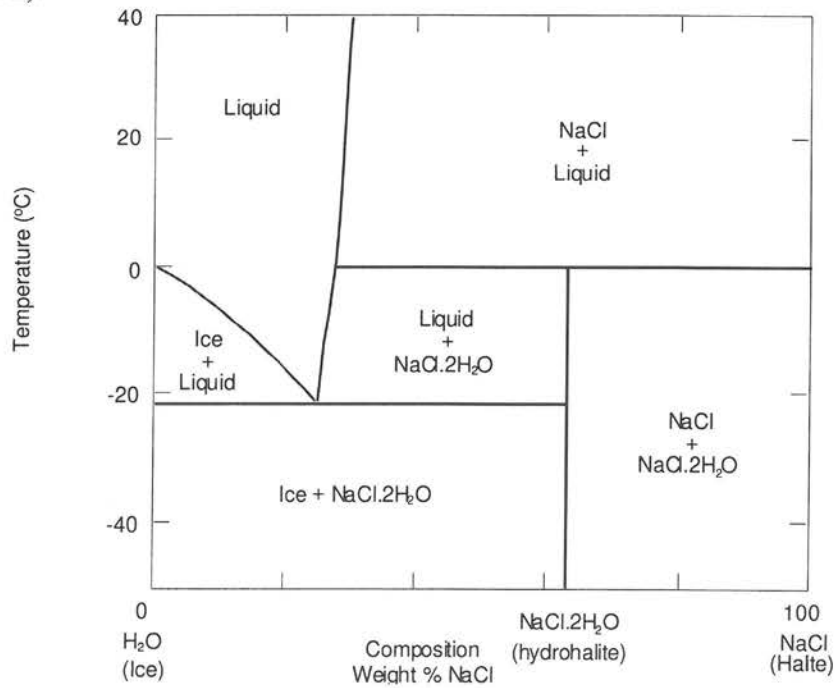
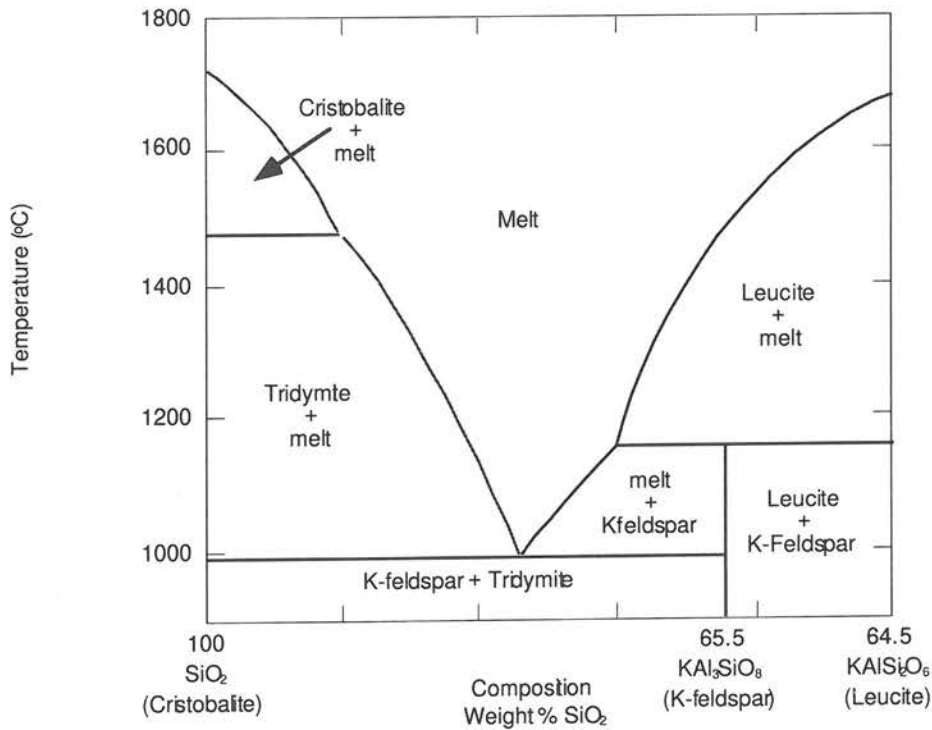


Figure 2c. The Leucite - Silica System. The composition scale is in weight percent SiO_2 . Note that the composition scale is truncated on the right). Redrawn from Schairer and Bowen, (1947).



weight. Such salty fluids are found in the Great Salt Lake and develop during the late stages of crystallization of igneous intrusions. (5) If a mineral contains a fluid inclusion in which the contained aqueous fluid has a eutectic temperature of -20.8°C and a final melting temperature of -4°C , then the fluid must have a salinity of approximately 3.5 weight percent NaCl. Seawater is such a fluid. Perhaps, the mineral precipitated from seawater! Many more things can be learned from this phase diagram, but this will suffice for now. Demonstrate to your instructor your understanding of phase diagrams by showing how each of the above real life applications can be deduced by inspection of Figure 2b!

The leucite - silica ($\text{KAlSi}_3\text{O}_6 - \text{SiO}_2$) system is relevant to study of the origin of certain igneous rocks, and the phase diagram closely resembles the ice-halite phase diagram (Fig 2c). The leucite - silica diagram tells us several things also: (1) Five phases are possible: the four minerals leucite, k-feldspar, cristobalite (quartz polymorph), tridymite (quartz polymorph), and melt of compositions ranging from KAlSi_3O_6 to SiO_2 . (2) The melting point of cristobalite and leucite (what are they?). (3) K-feldspar does not melt to a liquid of its own composition (it transforms to yield some melt and another mineral -- **incongruent** melting). (4) You will never find leucite and quartz in the same rock under equilibrium conditions -- a reaction will take place to form orthoclase under equilibrium conditions. (5) The vertical boundary at 65.5 weight percent SiO_2 is a fundamental dividing line in this simplified chemical system. Rocks containing greater than 65.5% SiO_2 will contain quartz whereas rocks containing less than 65.5% SiO_2 will not contain quartz-- a first order observation when you are trying to name a rock on the basis of its mineralogy!

The phase diagrams shown in Figures 2b and 2c are the result of considerable efforts by several experimentalists.

Table 1. Selected phase data for aqueous solutions of chloride species commonly found in fluid inclusions (from Crawford, 1981)

Dissolved Species	Eutectic T (°C)	Eutectic composition (wt.%)	Solid Phases	Solid Phases	Optical Properties	Solid Melting Relations
			H ₂ O	ice	hexagonal, colorless RI ε 1.313 ω 1.309	0 °C, congruent
NaCl	-20.8	23.3% NaCl	NaCl·2H ₂ O	hydrohalite	monoclinic	+0.1°C, incongruent
			NaCl	halite	cubic colorless RI 1.544	
KCl	-10.6	23.3% KCl	KCl	sylvite	cubic colorless yellowish RI 1.490	
CaCl ₂	-49.8	23.3% CaCl ₂	CaCl ₂ ·6H ₂ O	antarticite	hexagonal colorless RI ε 1.393 ω 1.417	30.08 °C, incongruent
MgCl ₂	-33.6	23.3% MgCl ₂	MgCl ₂ ·12H ₂ O			-16.4 °C, congruent
NaCl-KCl	-22.9	23.3% NaCl 23.3% KCl				
NaCl-CaCl ₂	-52.0	1.8% NaCl 29.4% CaCl ₂				
NaCl-MgCl ₂	-35.0	1.56% NaCl 22.75% MgCl ₂				

The Experiments

A portion of the phase diagram for the X - Y chemical system will be investigated by performing three heating and cooling experiments, A, B and C. Compound X is an unknown liquid at room temperature and compound Y is some unknown solid at room temperature). Small experimental capsules (either sealed transparent capillary tubes or synthetic quartz) containing random amounts of X and Y have been prepared. These samples were placed in an oven (the sample chamber of a Heating/Freezing stage) whose temperature could be precisely controlled and monitored ($\pm 0.05^\circ\text{C}$). The transparent nature of the experimental capsules permits direct observation of the contents during the experiment. As the temperature is alternately lowered and raised, changes take place (phase transitions-- melting, chemical reactions). Record the temperature at which phase transitions occur and note the identity of all phases present below and above the phase transition temperature for each reaction vessel. The instructor will lead you through a video recording of the experiments or show you a series of still frames that illustrate the progress of the experiment (experiment C only). You will make observations and collect requested data (see below).

EXPERIMENTS

In this exercise you will collect real data and construct a real phase diagram, and to add a bit of drama, even discover the identity of the chemical system you are dealing with. View the video of the three experimental runs. Answer the questions pertaining to each experiment at the conclusion of each experiment.

Experiment A

The experimental capsule is cooled until everything is solid (below -80°C) and then warmed slowly until the contents is entirely liquid. Describe what happens in detail.

Experiment B

The experimental capsule is cooled until everything is solid (below -80°C) and then warmed slowly until the contents is entirely liquid. Describe in detail what happens.

Experiment C

In this experiment we will make a sketch of the experimental capsule and measure the areas of the solid and of the liquid at various temperatures. The experimental capsule is cooled until everything is solid (below -80°C) and then warmed slowly until the contents are entirely liquid. At various temperatures we will make a quick sketch of the experimental capsule by placing a transparency on the TV screen and outlining the experimental capsule, the solid crystals and the gas bubble (think about how can the area of the liquid could be determined). There will be one sketch for each temperature. Each sketch will be distributed to a different student or group of students who will be responsible for determining the area of the inclusion (experimental vessel), the volume of the vapor bubble, and the volume of solid. This constitutes one data set. The area will be determined by placing the transparency on a sheet of graph paper and counting squares. Because the shapes are round, you may want to keep track of partial squares to get a more accurate count. Each student or group will be responsible for the data set corresponding their assigned temperature. When each student or group has finished making the area determinations for their temperature, the data will be tabulated on the board for everyone to record (Each student or group will supply values for one row-- Columns A-D; Figure 3). Be sure to record the temperature at which melting begins as accurately as possible.

From the area measurements we will determine the volume of the liquid, the fractions of liquid, and the salinity of the liquid at each temperature. The liquidus is plotted from the paired temperature - liquid salinity data (See next section).

QUESTIONS

Experiment A

- What type of melting behavior is exhibited by the contents of A (congruent or incongruent)?
- Based on your observations what must be in experimental capsule A (Refer to Table 1)?

Experiment B

- The melting behavior in *Experiment B* indicates that the experimental capsule contains at least two substances. The eutectic temperature can be used to uniquely characterize the chemical system for a mixture of two or more substances. What must be in capsule B (Refer to Table 1)?
- What factors might influence the accuracy of the eutectic temperature measurement? How precise do you think the measurement is?
- Potter (1978) in a series of experiments measured the “freezing point depression” of an aqueous fluid as a function of increasing amounts of various dissolved substances. He derived the equation below for your chemical system.

$$T = -0.581855*W - 0.00348896*W^2 - 0.0004314*W^3$$

$$W = -1.76958*T - 0.042384*T^2 - 0.00052778*T^3$$

where T = the final melting or liquidus temperature ($0 < T < -20.8^\circ\text{C}$)
and W = the final weight percent salt in the solution ($0 < W < 23$ NaCl wt % equiv.)

* What is the salinity of the fluid in capsule B?

Experiment C

- What substances does experimental vessel C contain?
- What is the salinity of the solution?
- The data in columns A- D can be used to experimentally determine the solvus in this system. To do this you need to first calculate the necessary values for columns E to G.

<u>Column</u>	<u>Header</u>	<u>Explanation</u>
A	Temperature	Temperature in $^\circ\text{C}$
B	Capsule area	Size of experimental vessel (area measurement in arbitrary squares)
C	Bubble area	Amount of bubble (area measurement in arbitrary squares)
D	Solid area	Amount of solid (area measurement in arbitrary squares)
E	Liquid area	Amount of liquid (area measurement in arbitrary squares)
F	XI	Liquid as fraction of total solid and liquid (ratio)
G	Liquidus	Wt. % NaCl Equiv. at temperature*

*At any given temperature a tie line (XZ in Fig. 2a) pairs the composition the solid with that of the liquid. The tie line XZ consists of the sum of the shorter segments XY and YZ, which represent the relative amounts of liquid and solid, respectively. Since the length of XY as a fraction of XZ is known (Col. F) the length of XZ can be determined if we know the absolute length of XY in units of weight percent ($XZ = \text{fraction liquid} / XY$). How can we determine the absolute length of XY in units of weight percent? One could do two experiments in which the freezing point of water is determined one that yields a temperature slightly higher and one that yields a temperature slightly

PHASE DIAGRAMS IN VIVO

Information for the Instructor

INTRODUCTION

The topic of phase diagrams in many mineralogy texts begins with an introduction to temperature-composition (T-X) diagrams (e.g. Klein and Hurlbut, 1993). Commonly the instructor is faced with introducing a plethora of terms (tie lines, joins, reaction lines, liquidus, solidus, melting point, incongruent, congruent melting, peritectic, eutectic, phase, equilibrium, disequilibrium, 1 and 2 phase fields) and explaining their meaning and significance to a by now bewildered audience. When students are able to visualize a phase diagram in action or even be engaged in creating one from their own experimental data, the vocabulary and concepts become intuitively clear. Fluid inclusions, which occur naturally in all minerals, are ideal experimental vessels that permit the acquisition of temperature - composition data for the construction of T-X phase diagrams. Fluid inclusions are particularly suitable because they bridge the gap between experimental petrology (mineral assemblages) and the minerals themselves (i.e. they give information on the conditions of formation of the minerals that contain them)!

This lab has been run using various formats depending on class size, time constraints, available equipment, and objectives of the instructor. If the class size is small, sufficient time is available and a fluid inclusion stage is available the entire class may visit the fluid inclusion laboratory to collect data in real time. For a large class or if a fluid inclusion laboratory is not available a video showing a laboratory tour and the experimental runs is used. Videos of the experimental runs were made by mounting a camera on a microscope and filming the changing proportions of solid (ice) and liquid (brine) in a synthetic fluid inclusion (in quartz) as it was cooled and heated in a FLUID INC. freezing / heating stage. A copy of this video can be obtained from the author by trade of a blank VHS cassette. A collection of still images from the video is also available and can be used in place of the video.

These images are available over the Internet at

<http://www.mines.utah.edu/~wmep/workshop/filab.html>

They can be used by individual students or as a group exercise for the entire class.

This exercise can be done as a homework assignment, although much of the value that the instructor can add would then be lost.

Three experiments have been videotaped. The first two are largely demonstrations, whereas students actively participate in the third experiment. The first experiment shows congruent melting of a substance at precisely 0°C. Students conclude that the substance in the experimental vessel must be pure H₂O as it is the only known substance showing this diagnostic behavior.

The second experiment demonstrates the reality of a eutectic point in a binary system, the significance of the eutectic point in characterizing the chemical system under investigation, and the fact that mixtures melt over a temperature range. This introduces the concepts of tie-lines, the lever rule, "freezing point depression", solid solution, and 1, 2 and 3 phase fields. Applications to everyday life (salting roads in winter) and magmatic systems are easy to point out.

The third experiment is the longest and most interesting. This experiment is essentially the same as the second except that we pause at various temperatures to make sketches to quantify the changing proportions of liquid and solid as temperature is raised. Although the required calculations can be done for each temperature individually on a calculator, it is useful to have the student carry out the calculation in a spreadsheet. Once the spreadsheet is constructed, graphing the results is straightforward. Errors and assumptions are easy to test. The final wrap-up can take one of two forms; an individual report to be turned in at a future time or an immediate group summary and discussion. If an overhead projection system is available for a lab computer the instructor can lead the discussion by suitably manipulating the data in real time to illustrate the discussion.

The purpose of this laboratory as designed is to gain familiarity with simple phase diagrams, their construction, and their applications to the understanding of geological and environmental problems. Subsidiary objectives include development of strategies for data processing including evaluation of assumptions and sources of errors, as well as honing of computer, spreadsheet, presentation (tabular and graphical), and report writing skills. This laboratory develops, reviews or makes use of the following concepts and/or skills.

EXAMPLES AND SOLUTIONS

Several questions are posed throughout the exercise. Suggested answers are provided below along with a real sample data collected during Fall quarter 1996 at the University of Utah (GG 308 Mineralogy).

Figure 1.

- Q.** What must the volume of the bubble be if the density of the vapor is 0.01 gm/cm^3 ?
- A.** At 250°C density = 0.8 gm/cm^3 (given). Assume a volume of 1 cm^3 . The mass of the fluid is therefore 0.8 gm . At 25°C the mass of the water and vapor together is 0.8 gm . The overall density must remain the same if the integrity of the fluid inclusion has not been violated. Furthermore, $V_f = 1 - V_v$. Therefore, $1.0 * V_f + 0.01 * V_v = 0.8$, $1.0 * (1 - V_v) + 0.01 V_v = 0.8$, $1.0 - 1 * V_v + 0.01 V_v = 0.8$, $-0.99 * V_v = -0.2$, $V_v = 0.2$ or about 20% of the volume of the inclusion!

Experiment A

The experimental capsule is cooled until everything is solid (below -80°C) and then warmed slowly until the contents is entirely liquid. Describe what happens in detail.

Liquid persists until the temperature drops below approximately -37°C . At that temperature the fluid turns into a very fine-grained solid. In this case the solid takes up more volume than the same mass of fluid and the bubble collapses a small but noticeable amount. Upon heating the solid recrystallizes to form a large clear crystal. As the melting temperature is approached noticeable changes in the morphology of the solid are seen. Melting takes place instantaneously at 0°C .

Qa. What type of melting behavior is exhibited by the contents of A (congruent or incongruent)?

Aa. *Congruent. Instantaneous melting at 0°C .*

Qb. Based on your observations what must be in experimental capsule A (Refer to Table 1)?

Ab. *The substance in capsule A must be pure H_2O (ice) as this is the only substance that has a melting point of 0°C .*

Experiment B

The experimental capsule is cooled until everything is solid (below -80°C) and then warmed slowly until the contents is entirely liquid. Describe in detail what happens.

Liquid persists until the temperature drops below approximately -37°C . At that temperature the fluid turns into a very fine-grained dark solid. In this case also, the solid takes up more volume than the same mass of fluid and the bubble collapses a small but noticeable amount. Upon heating the solid recrystallizes to form a large clear crystal. At about -20°C the solid begins to break up and liquid is visible. By -6.6°C all the solid has melted.

Qa. The melting behavior in *Experiment B* indicates that at the experimental capsule contains at least two substances. The eutectic temperature can be used to uniquely characterize the

chemical system for a mixture of two or more substances. What must be in capsule B (Refer to Table 1)?

Aa. *Capsule B contains H₂O and NaCl. The eutectic temperature observed near -20°C rules out the presence of KCl alone. Solution of H₂O and CaCl₂, MgCl₂, NaCl-KCl, NaCl-CaCl₂ and NaCl-MgCl₂ all have eutectic temperatures well below -20°C.*

Qb. What factors might influence the accuracy of the eutectic temperature measurement? How precise do you think the measurement is?

Ab. *Several factors can potentially influence the accuracy and precision of the eutectic temperature measurement. Instrumental factors: Rapid heating may register a temperature that is higher than actually present at the site of melting (disequilibrium). Observational: Ice float and because we are looking "down" the ice obstructs our view of any liquid that may be forming "below" it; The first "droplets" of liquid are likely to wet the solid and thus be difficult to discern; In dilute solutions the index of refraction of the liquid may be very similar to that of the solid making small amounts of liquid difficult to detect; In dilute solutions very, very small amounts of liquid will be produced initially (see Figure 2a).*

Qc. What is the salinity of the fluid in capsule B.

Ac. Use the relationship: $W = -1.76958 * T - 0.042384 * T^2 - 0.00052778 * T^3$, where T = the final melting or liquidus temperature ($0 < T < -20.8^\circ\text{C}$). For T = -6.6°C , W = 10.0 wt % NaCl equivalent.

Experiment C

In this experiment we make a series of sketches on sheets of overhead transparencies of the fluid inclusion as viewed on the monitor at various temperatures. The areas of the inclusion, contained solid, and bubble are determined by placing the transparency on a sheet of graph paper and counting squares (The area of the inclusion only needs to be sketched once because it does not change). The area of the liquid is obtained by subtracting the area of the bubble and the solid from the area of the inclusion. Applying the lever rule the locus of the liquidus can be calculated provided an absolute starting point is given. That starting point is the salinity of a solution whose freezing point depression is -6.6°C .

Qa. What substances does experimental vessel C contain?

Aa. *Same answer as Experiment B.*

Qb. What is the salinity of the solution?

Ab. *Same answer as Experiment B.*

REQUIRED MATERIALS

- Video of a laboratory tour and an experimental run. Alternatively, still images of the experiment as it proceeds. Experiment A cannot be done with still images alone. The same images used in Experiment C can be used for Experiment B. The lab description assumes that a video or stills are being used in the lab.
- TV monitor/VCR to play back video recording of experimental run or for live data collection.
- Overhead transparencies, marker pen, and graph paper for quick area determinations.

ACKNOWLEDGMENTS

The exercise was considerably improved by the comments of John Brady, Dexter Perkins, Andy Campbell, Marlene McCauley. Feedback from the attendees of the Mineralogy Workshop was much appreciated.

Figure 3. Sample Data Table (Columns A - D are for data; Columns E - G are for calculated values) The values in columns B, C and D are area measurements (squares on a sheet of graph paper). The absolute values depend on the size of the squares and also on the size of the image on the monitor! The calculations for columns E - G are explained in the student hand out.

A	B	C	D	E	F	G
	1257					-6.6
Temperature	Capsule Area.	Bubble Area	Solid Area	Brine Area	XBrine	Liquidus
-18.0	1257	239	553	465	0.46	21.9
-17.0	1257	248	494	515	0.51	19.6
-16.0	1257	251	505	501	0.50	20.0
-14.0	1257	264	428	565	0.57	17.5
-13.0	1257	273	412	572	0.58	17.2
-12.0	1257	263	382	612	0.62	16.2
-11.0	1257	277	345	635	0.65	15.4
-10.0	1257	295	298	664	0.69	14.5
-9.0	1257	290	197	770	0.80	12.5
-8.0	1257	276	133	848	0.86	11.6
-7.5	1257	293	53	911	0.95	10.6
-7.0	1257	311	11	935	0.99	10.1
-6.6	1257	322	0	935	1.00	10.0
Salinity	Temp					
10.0	-6.6					

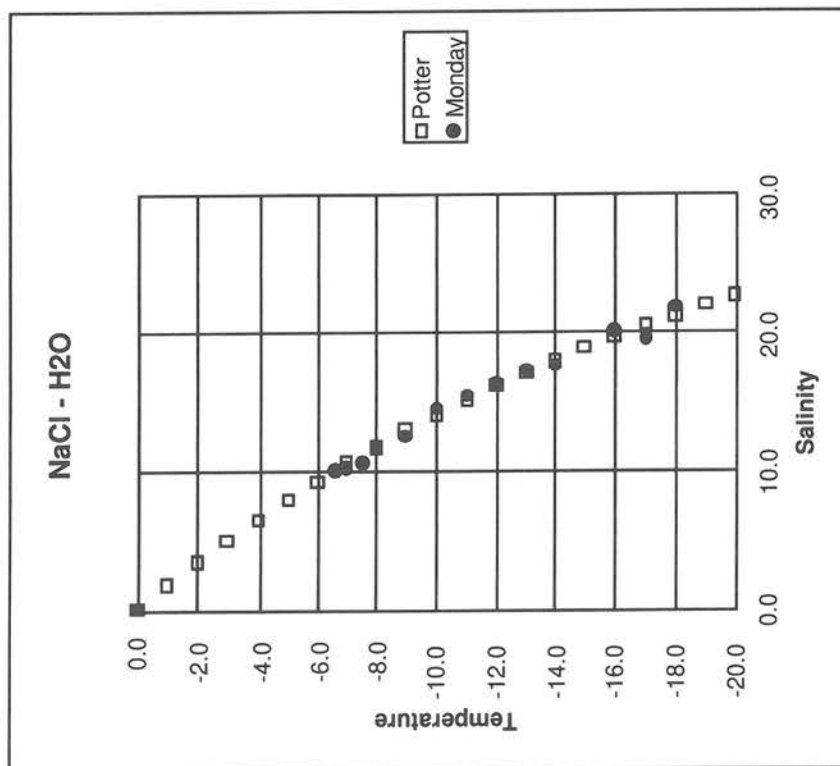


Figure 4. Temperature (A) vs Liquidus composition (G)