PVT Measurements and Properties of a Simple Compressible Substance

Read this document before lab. You may skim apparatus description and procedures until you actually get into lab. Get the "big picture". We have given you an outline in Appendix A of what is expected in a lab report. Appendix B is a brief discussion of LabView, which you may need to refer to during the experiment, but there is no need to read in advance. Appendix C is a definition of specific heat and latent heat: read it before coming to lab so you know what you're measuring. Appendices D, E, and F describe how temperature sensors, heat flux sensors, and pressure sensors work. Glance at them beforehand to get the idea of how they work; you may have to pay careful attention to how the heat flux sensor works as this is an uncommon device. You may skip the technical specs until you need them.

Wear to lab: Appropriate clothing that you won't mind getting dirty; NO OPEN-TOE SHOES
Location: Pierce G11 for the first half; MD B133 for the second half.
Bring to lab: A calculator; this document; course text and bring-to-class binder.

In this laboratory we will introduce standard methods and devices for measuring three basic thermodynamic properties of a simple compressible pure substance: pressure, volume, and temperature. In the first section we will measure the thermal history associated with the cooling and solidification of a molten metal. In the second section, we will measure the equation of state (the relationship between pressure, volume, and temperature) describing the liquid and gas phases of a simple compressible pure substance, and calibrate with a measurement of atmospheric pressure. The objectives of the laboratory are:

(i) To familiarize you with hardware commonly used for measuring T, P, and V.
(ii) To explore the properties and phase change behavior of a simple compressible pure substance and compare them to what you have heard/read about in class.
(iii) Introduction to how to measure heat transfer.

Equipment List:

Pressure measuring devices: manometers, Bourdon tube gauges, and pressure transducers will be available for your inspection. Before you come to lab, review your course text (Sonntag, chapter 2) for a description of how a manometer works, and see Appendix F of this lab handout for a description of how a pressure transducer works. The principle of operation of a Bourdon gauge will be explained during lab.

Temperature measuring devices: alcohol and mercury thermometers, bimetallic devices, thermocouples of various types, thermistors, and an infrared pyrometer will be available for your inspection. Before you come to lab, see Appendix D for descriptions of how thermocouples and thermistors work. See the next page for a sufficient description of the bimetallic device.
**Volume measuring device:** graduated cylinder (rated for high pressure gas containment).

**Other equipment:** A heat flux sensor. Before you come to lab, see Appendix E for a description of how this works.

**Part I: Temperature measurement and Calorimetry**

**A. Temperature Measurement**

You will be able to observe in lab the following standard devices:

(i) **Thermocouples:** A potential difference exists at the interface of two dissimilar metals (this physical response is called the *Seebeck effect*). This potential difference (or *electromotive force*) is a function of temperature and thus a measurement of this potential difference allows a measurement of temperature. A 'cold junction' at a known, calibrated, constant temperature must be provided either mechanically with an ice bath or electronically with a conditioning circuit that corrects for variations in room temperature.

(ii) **Bimetallic devices:** Two pieces of metal with different thermal expansion coefficients are bonded together to make an initially straight strip. This strip is deflected when the temperature changes to a temperature different than the bonding temperature. The curvature of the strip is proportional to the change in temperature.

(iii) **Thermistors:** In these semiconductor devices the electrical resistance decreases as the temperature increases (for most semiconductors resistance *decreases* as the temperature increases unlike metals whose resistance *increases* as the temperature increases). For this particular thermistor the governing equation is

\[
\text{Resistance [in Ohms]} = (-32.402)(\text{Temp. in } ^\circ\text{C}) + 4593.39
\]

(iv) **Liquid filled thermometers:** Thermal expansion of liquid in a closed glass capillary indicates the temperature.

(v) **Infrared pyrometers:** Focuses the infrared radiation from the measured surface onto a temperature-sensing element which uses an average value for the emissivity (the emissivity provides a quantitative measurement of the ability of a surface to radiate energy; see Sonntag chapter 4).

**B. Calorimetry**

(i) **Introduction:**

In many of the homework questions you are asked to use values of thermodynamic properties of pure materials that must be experimentally determined. The first such measurements were made by Lavoisier and Laplace from 1779 to 1789 as part of the development of the Caloric Theory, and the science of making reliable observations of such properties is now called *Calorimetry*. 


Since the temperature changes are often quite small, accurate experiments require very elaborate and methodical experimental techniques. Modern calorimetry experiments are usually carried out in a Differential Scanning Calorimeter, or DSC, which monitors the heat flow and temperature of a small sample of test material relative to that of a control sample, thereby permitting very high precision.

(ii) Objective:

In this part of the lab, you will make simple, direct measurements (without a control sample, so not highly accurate) of the temperature and heat flow from a molten sample of tin (Sn) alloy as it cools and transforms from the liquid phase to the solid phase. You will thereby estimate the heat capacities in the liquid and solid phases and also the latent heat of fusion. Explanations of the heat capacity and latent heat of a material can be found in Appendix C.

(iii) Details:

You are provided with a type "K" (chromel/alumel) thermocouple having a suitable operating range such that it can be immersed in the molten metal sample. You are also provided with a solid-state thin film 'heat flux' sensor (a thermopile transistor encased in an inert film), which generates a voltage proportional to the rate of heat flow per unit area through the sensor (SI units for heat flow are J/(m²s)). The sensitivity of this particular device is given in Appendix E, and in order to amplify the small voltage generated by the sensor, the output is filtered and amplified by 1000X using a low-noise preamplifier.

CAUTION: The heat flow sensor is encased in an orange polymeric film called 'Kapton' which is reasonably heat resistant (i.e., won’t decompose), but the sensor is FRAGILE and should be handled CAREFULLY! It should NEVER be put into contact with the liquid metal sample.

DANGER: The liquid metal is very hot! Exercise care not to spill on you (or your neighbor). ALWAYS wear safety goggles and gloves (these will be provided in lab) when handling the sample.

1. Before heating the sample, extract the solid tin from the ceramic crucible and weigh both the sample and the crucible. Also, measure the surface area of the heat flux sensor window (the entire area through which heat can flow).

Both the thermocouple and the heat flux sensor provide voltages proportional to the quantity being measured. These voltages can be monitored directly using multimeters or by using the LabVIEW software.

NB: (a) Unlike the thermocouple, the heat flux sensor is not electronically compensated and you must manually 'zero' it by measuring the mean voltage generated by the sensor when it is sitting at equilibrium (no net heat flux) on the lab bench.

(b) Also note that the output of the device is weakly temperature dependent. A type K thermocouple is integrally mounted within the Kapton substrate of the sensor, and by monitoring the temperature of this sensor, this effect can be corrected for using the calibration curve given in Appendix E.
(c) By GENTLY placing your finger on either side of the film, note what happens to the magnitude and sign of the voltage generated by the sensor. Be sure to let the sensor approach steady state before measuring the heat flux. For those of you who have taken AM105b or a course in heat transfer, you may note that the transient response of the device to a step change in heat flux could be modeled by solving the appropriate thermal diffusion equation.

2. Heat the sample in the crucible using the provided propane torch to at least 300 °C (if your sample is pure Pb, heat to at least 350 °C), at which temperature the sample should be completely molten. At that time turn off the torch, remove the crucible from the holder with tongs, and place it on the cork crucible holder.

3. Quickly lower the thermocouple into the liquid metal and carefully place the heat flow sensor over the top of the crucible in order to avoid heat loss through any surface but the top one.

4. The heat flux out of the crucible (and thus across the sensor) is very sensitive to air currents which leads to convection of heat. To minimize convective currents, a shield (in this case a cardboard box) should be placed around the crucible/sensor setup. If convection is "shut off", what other mechanism(s) of heat transfer must be responsible for the flow of heat out of the molten tin?

5. Run the LabVIEW file "TIN.VI" to monitor the sample temperature $T(t)$ and the heat flux $\dot{q}(t)$ through the sample as a function of time. Run the program for about 12–15 minutes. The VI will also monitor the temperature at the surface of the heat flux sensor (in order to correct the heat flux reading relative to temperature as Appendix E) The file voltage data are listed as:

   Heat flux sensor temperature, tin temperature, Heat flux.

   Temperature is in millivolts, i.e. 100mV = 100 °C. The heat flux has 100X amplification.

6. With your lab partners discuss how you can use your knowledge of $T(t)$ and $\dot{q}(t)$ together with the First Law of Thermodynamics (plus the knowledge of the sample mass, which you should make sure you determine, and area of the top of the sensor surface) to calculate the following quantities:

   • the specific heat of liquid tin: $C_p^{\text{liquid}}(T)$
   • the specific enthalpy of fusion for tin: $h_{\text{if}}$
   • the specific heat of solid tin: $C_p^{\text{solid}}(T)$

   Carefully list all assumptions you make in your analysis!

7. Compare your answers from part (6) with the accepted literature values for tin which you can find in the CRC Handbook of Chemistry and Physics or Marks' Handbook for Mechanical Engineers.
Part II: Pressure measurement and PVT behavior

A. Barometric pressure

We have defined the pressure exerted by the ambient air on any surface as 'atmospheric pressure'. The local value of atmospheric pressure depends on several factors including the temperature, elevation, and wind speed. By definition, standard atmospheric pressure corresponds to the pressure at the base of a column of 760 mm of mercury (Hg) at 0 °C and with g = 9.807m/s². Thus,

Standard atmospheric pressure = 760 mm Hg = 14.7 lbf/in² (psia)=101.325 kPa = 1.01325 bars

Question: Determine the height of a water column with a pressure of 1 atm. at its bottom and 0 at its top.

Find today's atmospheric pressure using the following devices:

(i) Mercury barometer: Fortin-type which is located on the wall by the door to the lab. Instructions are on the device. Make the appropriate correction for temperature.

(ii) Access the campus computer network and determine the atmospheric pressure by pointing your browser to http://www.weather.com/weather/local/02138

In both cases convert values of the local atmospheric pressure to inches of mercury, standard atmospheres, and kilopascals. Compare the values in your laboratory report.

B. PVT behavior

This high-precision experimental apparatus is used to investigate the equation of state of the liquid and vapor phases of a pure substance in the vicinity of the saturation dome. The working fluid, sulphur hexafluoride (chemical formula SF₆), is chosen for its convenient critical point (Tᵥ = 45.56 °C; Pᵥ = 3820 kPa). The actual device consists simply of a gas and mercury filled glass cylinder with the volume of fluid above the SF₆-Hg meniscus calibrated by markings along the side of the cylinder. Changes in volume may be controlled by a hand wheel that moves a piston fitted to the cylinder. A constant temperature bath circulates water around the glass measuring tube, which is enclosed within a transparent water-filled chamber, allowing precise control of the temperature.

The PVT behavior is investigated by recording the isothermal lines on a pressure-volume (PV) plot and observing the appearance and disappearance of an interface between vapor and liquid SF₆ as the fluid is compressed at constant T. Using these observations and noting changes in slope in the measured isotherms, and repeating these observations at several temperatures, ultimately the saturation dome between liquid and gaseous state can be obtained. This diagram should show the distinction between the vapor, liquid, and saturated mixture regions on the PV diagram.
The circulation bath

First check the level and the temperature of the water in the Lauda E-100 circulation bath. Make sure the level is adequate: the black plastic intake should be submerged so that the pump will not cavitate. Add water or ice if necessary (note: your first readings will be at the lowest temperatures, so adding ice may be more appropriate). Make certain the rubber plug with the yellow type K thermocouple at the top of the water jacket viewing magnifier is tightly capped so that the pressure generated by the rubber tubing doesn't spew water out of the water jacket! Become familiar with the button controls on the front panel display of the Lauda circulation bath. You may select a bath temperature with 0.1 °C precision on the digital display by scrolling up or down using the [^ / v] buttons and selecting the desired temperature with the [● >] button. The speed of the flow may also be altered; low 1 to hi 5 [ run at maximum speed ] with the [● >] button.

Temperature measurement and steady state

For the simplest analysis this particular experiment requires (as do most in thermodynamics) steady-state to be attained before measurements are made. Here, water is circulated at some preset temperature and the mercury ‘fin’ pressurizing the gas within the glass tube is heated to the desired temperature within the water jacket. When the temperature is stabilized, data may then be collected. A yellow type K (chromel/alumel) thermocouple will be used to monitor temperature within the water jacket. Turn on the Omega TAC30K thermocouple to millivolt converter and turn on the multimeter, setting it to read DC millivolts. Manipulate the thermocouple probe through the rubber stopper so that the tip rests against the top of the glass cylinder containing the SF6. This will enable an accurate determination of the temperature of the water bath where it surrounds the gas.

The pressure transducer

The apparatus uses an Ashcroft gauge pressure transducer made by Dresser Instrument, a compact stainless steel explosion-proof sensor that runs on 10 – 36 VDC. The transducer has a range of zero to 1000 psi gauge. - with a corresponding output voltage of 1 to 5 VDC and an accuracy of 0.5 %. (e.g. a 2.00 VDC reading = 250 psig. Note that the output is in English units - conversion factor: 1 p.s.i. = 6.895 kPa).

The power supply

Twelve volts DC is a good choice to power the pressure transducer. Turn on the Agilent E3631A Triple Output Power Supply. It should say ‘output off’. Select ‘+25”’. Select ‘Display Limit’. Turn the knob on the upper right face panel to get +12V displayed and finally the ‘output on’ button. Turn on the Agilent 34401A Multimeter. It should display a value in DC volts representing the transducer pressure measured within the apparatus.

Absolute pressure
As pressures for PVT diagrams are always reported in absolute terms, the gauge pressure readings must be converted to absolute pressure readings for the desired final PV plot. This may be accomplished thus: Read the current local barometric pressure in mm of Hg on the Fortin type barometer in the ME Lab, or use some other valid local reading. Record it in your notes. Convert barometric pressure into kPa (conversion factor: 101.325 kPa = 760 mm Hg). This offset must be added to the transducer’s gauge pressure readings to obtain values of absolute pressure.

Procedure

After final connection to the isothermal bath, the temperature is adjusted to the appropriate value. [See 'Recommended temperatures' below.] When it has becomes stable, data are taken; and then the mercury column is raised to the next increment following the volume graduations recommended further below. Turning the handwheel moves the internal piston and changes the internal volume of the cavity. The maximum volume that may be measured is 4 ml, with 0.05 ml graduations. The corresponding pressure may be read from the transducer in DCV at the value of volume selected.

SAFETY NOTE: As you reduce the fluid's volume, never let the pressure transducer's output exceed 4 volts. Monitor the pressure transducer with special care when the fluid is reduced in volume to near its minimum: the isothermal compressibility gets small, meaning small volume changes are accompanied by great increases in pressure. The stored internal potential energy within a highly compressed fluid can be enormous, and raising the pressure too high risks an explosion! When finished with all data collection, return the mercury level to its lowest level; essentially reducing the pressure to a very low value.

To eliminate possible artifacts due to “backlash” in the system, always make the final volume adjustment a decrease of the volume before taking readings. As data from point to point is collected, be sure to allow time for the pressure to ‘stabilize’ at each recording, which may be as long as 20-30 sec per data point. When you reduce the volume by turning the hand-wheel suddenly, you are doing nearly adiabatic work on the SF₆ thereby raising its internal energy and making it slightly hotter. It takes some time for heat to leak out through the glass tube to the temperature bath, thereby re-establishing equilibrium. The thermocouple is too far away from the SF₆ to record these temperature changes, but you may observe the effect: as heat leaks out of the SF₆ and its temperature drops at constant volume, its pressure also drops.

If the temperature is held constant below the Tc, we expect the gas begins to condense as the volume decreases, passing into the saturation dome. We expect the pressure to remain constant for a finite range as we move along a "tie line" within the saturation dome.

PV isotherms should be recorded for several different temperatures. After converting the pressure to absolute kilopascals and the volume to ml, all isotherms should be plotted on the same PV diagram. In reading volumes in a graduated cylinder, use the height of the center of the Hg column, not the height of the edge.

Recommended temperatures and graduations
Try these temperatures. We may change them, depending on how many lab groups we actually end up with.

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Finally, at your highest T, go out to the largest possible volumes and measure a small portion of the PV curve. Under these conditions the gas must be quite close to ideal (see the Lee-Kesler compressibility chart to figure out how close), and the mass can be determined with the least uncertainty from these readings.

After steady state has been reached, start at a maximum volume of 3.0 ml and take data while decreasing the volume, i.e. compressing. For starters, try these volume increments. (You may want to do another isothermal run or take some intermediate points in the vicinity of the boundary of the saturation dome):

- When the absolute volume is above 2.0 ml, use increments of 0.5 ml.
- When the absolute volume is between 2.0 and 0.5 ml, use increments of 0.1 ml.
- When the absolute volume is below 0.5 ml, use increments of 0.05 ml.

**Recommended procedure:**

1. Decrease volume rapidly (1-2 sec) and then wait for perhaps 20-30 sec while the pressure stabilizes.
2. Record: $T \ (^\circ C)$, Vol (ml), $P_{gauge}$ (Volts), phases.

Note the production of “rain” when you decrease the volume sometimes, and “boiling” when you increase the volume sometimes. The first appearance of this phenomenon signals a slope change in the isotherms.

Note the existence or absence of a visible interface between liquid and vapor. As the critical point is approached, any difference in the refractive indices of the phases gets smaller and smaller, and therefore harder and harder to observe. Near the critical point, you may want to test for the existence of an interface by making sudden, very small, increases and decreases in volume and watching for "rain" or "boiling" within a portion of the SF$_6$. Watch the interface to see whether it vanishes during the time over which the system is equilibrating thermally.

**Data conversion and P-V plot:**
Make additional columns for $P_{\text{gauge( psig)}}$, $P_{\text{gauge(kPa)}}$, $P_{\text{absolute(kPa)}}$ and $Z$ in the data chart. 
To convert volts to psig, subtract 1 and multiply by 250. 
To convert psig to kPa(gauge), multiply by 6.894757. 
To convert kPa(gauge) to kPa(absolute), add your barometric pressure reading, which should be somewhere in the vicinity of 100 kPa.

Plot your data by hand (volts vs. volume if you can’t convert quickly; otherwise absolute pressure in kPa vs. volume), as you take it, on graph paper in order to see where you are. Convenient axis limits are 0 to 3 ml (long axis, horizontal) and 1.5 to 3.5 volts or 900 to 4500 kPa (short axis, vertical).

Please note: the sets of curves are recorded from the right to the left in the PV diagram.

In your report, provide a plot of the P-V diagram along with the isotherms measured by your group and by all the other groups that have taken the lab within 48 hrs of the time yours is due. Indicate the saturation dome.

**Analysis:**

Determine the mass (in kg) and number of kmol of SF$_6$ in the apparatus.

Determine the compressibility factor for each data point and plot your data superposed on the Lee-Kesler (from your textbook) and Nelson-Obert (from the handouts) generalized compressibility charts. How well does this substance conform to the principle of corresponding states? Discuss possible sources of disagreement. For which of your data does the ideal gas equation of state work best, and how big a percentage error does it make in predicting v from P and T at that point?
Appendix A: Notes on your Laboratory Report

Your lab report is due by 5pm one week after the day you perform the lab. Please indicate on the front page of your report the date and time you took the lab.

In general, I do not expect lengthy laboratory reports but I do expect them to be neat and well presented (preferably typed).

Your report should read like a brief ‘technical communication’ in a scientific journal – it does not have to be long, but it should be complete and self-contained with appropriate references. You may list the lab handout as a reference but be sure to carefully note any errata or changes in procedure that you may have employed. Give complete references to any other textbooks or material you cite.

The best way to think of the report might be as follows: Imagine you are an engineer in industry writing a ‘Technical Summary’ of the experiments that is to be passed on to upper management. The presentation must be succinct enough that it will be read (!) but scientifically complete enough that strategic decisions can be made on the basis of the data you provide.

The following are guidelines in creating what will be considered a “complete” report.

Introduction:
As a matter of principle, please include a brief introduction that would orient someone unfamiliar with the lab as to what you’re doing. This needn’t be lengthy, but should give a clear idea of the lab’s purpose.

Procedure:
As the procedure is outlined clearly in the lab handout, it is not necessary to repeat too closely each step that was performed. Simply give a short overview of what you did to accomplish the purpose of the lab. There are questions in the procedure part of the handout; it is not necessary to answer these in your write-up, but they may guide your thinking in what to take note of.

Equipment:
Briefly discuss the major pieces of equipment used in the lab, as pointed out in the lab handout or by the TF. Explain their purpose in the lab and, if applicable, any deviations from the ideal.

Data:
Include that data that you collected in the form of printouts or notes where appropriate.

Analysis and discussion:
This will be the longest and most significant part of the lab report. Show your analysis of the data, including significant equations (Microsoft Word’s Equation Editor is helpful in inserting these). Include charts and graphs as appropriate. Discuss possible sources of error. It
is not necessary that you get causes of error exactly correct, but show that you understand what major factors may be causing deviation from the ideal case. Answer questions completely, explaining how you got your answers. Where appropriate, explain their significance in a broader sense.

Conclusion/Summary:
Give the take-away message of the major concepts of the lab.
Appendix B: LabVIEW

Variables altered during an experiment may be plotted as a function of time using a PC with the data acquisition software package LabVIEW. This is accomplished by monitoring the voltage generated by the sensors connected to an analog (real world) to digital (computer) "breakout" board. The program files for the experiment should be loaded and ready to run when you enter the lab. The TF's will be available to assist you during the experiment, however the following section contains a very brief description of the keystrokes necessary to perform a 'run'.

(With the VI on the screen for reference:) You may clear any data plots that already exist by clicking on the graph/chart of interest with the right mouse button and then highlighting the option "Data operations". A popup menu will appear; highlight "Clear graph/chart" with the right mouse button. Note: if the VI has a chart buffer, move the "Chart history size" or "Acquisition time" slide/value by clicking on it and altering it's value with the LEFT mouse button to change the size of the chart. Otherwise, the previous plot on the display will reappear at the beginning of the next run.
Appendix C: Specific heat and latent heat

The specific heat, (synonyms: "heat capacity", "specific heat capacity") of a material held at constant volume is defined as:

\[ C_v = \frac{1}{m} \left( \frac{\partial U}{\partial T} \right)_v \]

while the specific heat capacity at constant pressure is given by:

\[ C_p = \frac{1}{m} \left( \frac{\partial H}{\partial T} \right)_p \]

(Note: "specific" means "per kilogram" or, when we are using mole-denominated units instead of mass-denominated units, "per mole"). However for liquids and solids \( dH \approx dU \) (Sonntag, §5.6) so the specific heat capacities at constant pressure and constant volume are also approximately the same i.e. \( C_v = C_p = C \). Thus we have that:

\[ dU = m C dT \]

and assuming that the specific heat is independent of temperature (a good assumption over small temperature ranges), we can extend this expression to finite temperature changes as follows:

\[ \Delta U = m C \Delta T \]

Thus, in processes in which negligible work is done (such as the one in today's lab) we have that:

\[ \Delta U = Q = m C \Delta T \]

In contrast, the latent heat refers to phase changes at constant T. When a solid is heated to its melting point, no further temperature change will occur until the phase change to liquid is complete despite the fact that heat is being added. All of the heat goes into the phase change. The amount of heat needed to melt (or solidify) one kilogram of material is called \( h_{if} \), the specific enthalpy of fusion or, for short, the enthalpy of fusion or the latent heat of fusion or the heat of fusion. This quantity is related to the heat transferred as follows:

\[ Q = m h_{if} \]

There is a quantity \( h_{fg} \) defined similarly for the liquid-vapor phase change, called the specific enthalpy of evaporation, the enthalpy of fusion, the latent heat of fusion, or the heat of fusion. Using the saturation tables in your book, \( h_{fg} = h_g - h_f \) at a given temperature and pressure.

Note also the appearance of the analogous quantity for solid-vapor phase changes (sublimation), \( h_{if} \), in Table B.1.5 of your text.