AMMONIA DENSITY MEASUREMENTS

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ABSTRACT:

Over the course of this semester we successfully measured the density of frozen NH_3 and ND_3. We performed the measurements at liquid nitrogen temperature and dry ice temperature. When we compared our density measurements to accepted values we had an error of under 2%.

Introduction:

There are several methods used to measure density. Through X-ray diffraction techniques one can determine the density, if the lattice constants are known. However, without an X-ray diffraction machine equipped to handle materials which melt at negative 60 degrees centigrade, the easiest method for measuring density is the Archimedes displacement method. The mass of an object is easy to measure and the volume is determined by the amount of liquid displaced by the object. As NH_3 and ND_3 melt at -60C, it is necessary to find a substance that has a well determined density and remains a liquid at below -60 C. The liquids available to us were liquid nitrogen, which boils at 77K, and methanol, which remains a liquid at dry ice temperatures 195K. In the experiment we used both of these substances.

To measure density we needed a flask we could use to measure volume accurately and a scale that we could measure mass. The flask we chose was a standard chemistry flask with gradations from 100 to 110 ~~centi~~milliliters, with markings every tenth milliliter. We chose a standard digital scale that measured to centigrams. The flask was placed in a dewar thermal flask and the outside was

filled with either liquid nitrogen or dry ice methanol.

There is one problem with this technique and that is the nitrogen or dry ice that surrounds the flask evaporates from the dewar. To perform a displacement the dry sample is placed in the flask and weighed. Then the liquid is placed in the flask and the volume and weight are measured. Knowing the density of the liquid and the change in weight when the liquid was added, we can determine the volume of the liquid. As we know the volume of the total, we can determine the volume of the mass by subtracting the liquid volume. With the mass and the volume the density is easily measured. However, if after placing the sample the mass of the dewar changes because of evaporation, we cannot accurately calculate the volume of the liquid from the change in mass of the total. The change of mass of the dewar is most pronounced using liquid nitrogen, but it is also quite noticeable with dry ice.

To remedy this situation we use a computer to determine the mass continuously. To make a measurement the computer records the mass every second, once we know the mass of the dry sample we loaded the liquid and waited for it to stabilize. Looking at the change in mass as a function of time caused by the evaporating liquid we can extrapolate back to determine the mass of nitrogen in the dewar when we loaded the liquid. For the determination of the change in the total we use the mass in the dewar at the time of loading. For measurements made with methanol there is only evaporation from the dewar, but for the nitrogen there is some evaporation from the flask. Fortunately, this does not change

the measurement because we measure the volume, which compensates for the losses in the flask. We used a 486DX running at either 16 or 33 megahertz to take and process the data. The data acquisition program was the spread sheet As-easy-as version 5.0.

The volume of the flask is compensated also because of the contraction of the glass. The flask was calibrated for 20C and the change in temperature results in a significant change in volume. The glass is Kimax and has a well known contraction coefficient and allows an accurate measure for the change in volume.

Background Checks:

In order to determine if the linear extrapolation would be a reasonable one, we measured the evaporation from the dewar and the dewar and flask assembly as a function of time. The evaporation rates are just what we would expect. When the flask is first filled the nitrogen in the neck evaporates quickly as it cools the flask and is non-linear. As soon as the flask is cool, the evaporation rate stabilizes and it quite linear over the pertinent range.

In order to measure the density of the solid the density of the liquid needs to be known accurately. For methanol the density at dry ice temperature is easily measurable using our apparatus. For liquid nitrogen the density is measurable with our apparatus, but the accepted density is only known to about 0.5% error. As we intent to have an error measurement for the

frozen ammonia of 1-2%, 0.5% is a substantial contribution. We verified the density of liquid nitrogen using our apparatus.

One background check we performed without intending to was the check that none of our liquids reacted with our solids. Liquid nitrogen does not react and the ammonia is stored in liquid nitrogen. We were confident it would not react. For the first measurement using methanol we cooled the methanol by placing chunks of dry ice in it. Unfortunately, some of the dry ice dissolved in the methanol and when it came in contact with the frozen ammonia a reaction occurred. The heat of the bath increased sharply and the frozen ammonia went into stable solution with the methanol. The next time we cooled the methanol, we placed it in a flask and then placed the flask into a dry ice bath. When the ammonia was placed in the methanol this time, there was no reaction. The dissolved CO_2 changes the density of the methanol, for the accuracy of the measurement it is important that the methanol is pure.

Procedure:

The following procedure was determined over the course of a semester by trial and error:

1.) In the beginning the apparatus is cooled. The inside of the flask is filled with either nitrogen or super cooled methanol. While the apparatus is cooling, the samples can be retrieved from the storage dewar. Cooled fluid is placed in a dewar that pours

easily for use later.

2.) At this point the computer is started and we verify that the data is regular: one data point per second with a minimum number of discontinuities.

3.) For ease in loading, the samples should be taken from the small sample containers and loaded into one larger container. This makes loading easier and minimizes the chance that ammonia will stick to the side of the apparatus during loading.

4.) Now we are ready to take data. The flask is carefully emptied, replaced, and the scale tared. The sample is then loaded into the flask with a cooled funnel.

5.) The scale is tared again and the liquid is loaded into the flask. If the liquid is nitrogen it should be loaded up to the middle of the funnel, because evaporation will bring the level down. If the liquid is cooled methanol, it will not evaporate and the level should be raised to one convenient for making volume measurements.

6.) Now the apparatus is allowed to stabilize and we wait until the liquid in the flask stops boiling so that we can read the volume accurately.

7.) To read volume, the flask is lifted and a flashlight shown from behind. The flashlight makes the level of the liquid easier to see. Sometimes humidity from air condenses and makes the volume more difficult to see. Lifting the flask changes the mass considerable and when the data is plotted on the computer the points that we made volume measurements are easily seen.

8.) Several volume measurements are taken to find a good average. The measurements are separated by a few minutes.
9.) The data acquisition is now halted and the samples are loaded back into the storage containers. The data is converted from the format of the scale output to more useful numbers.
10.) The data is now graphed and analyzed to determine density.

Analysis:

At the end of this report there is a graph from a data run. From the graph it is not difficult to see what step of the procedure is being performed. At the beginning, the mass is loaded and the mass seems to decrease linearly. As the mass in the dewar decreases because of evaporation the mass appears to decrease. The mass we use for our calculations is the mass of the sample plus the nitrogen lost. We calculate the mass by fitting the mass weight curve to a linear regression and the point that this line crosses the time we started to load sample is the mass. Next we tared the scale and the mass goes to zero. When we start to load liquid onto the sample, the liquid evaporates and boils. As can be seen from the graph, the rate of loss is not linear but quickly stabilizes and becomes linear. When we make a volume measurement we fit the points right before the measurement and plot the line back to the point we started adding liquid. The mass of the liquid loaded is the point the straight line crosses the loading time. We now know the mass of the liquid loaded and the volume of the total. Since we know the

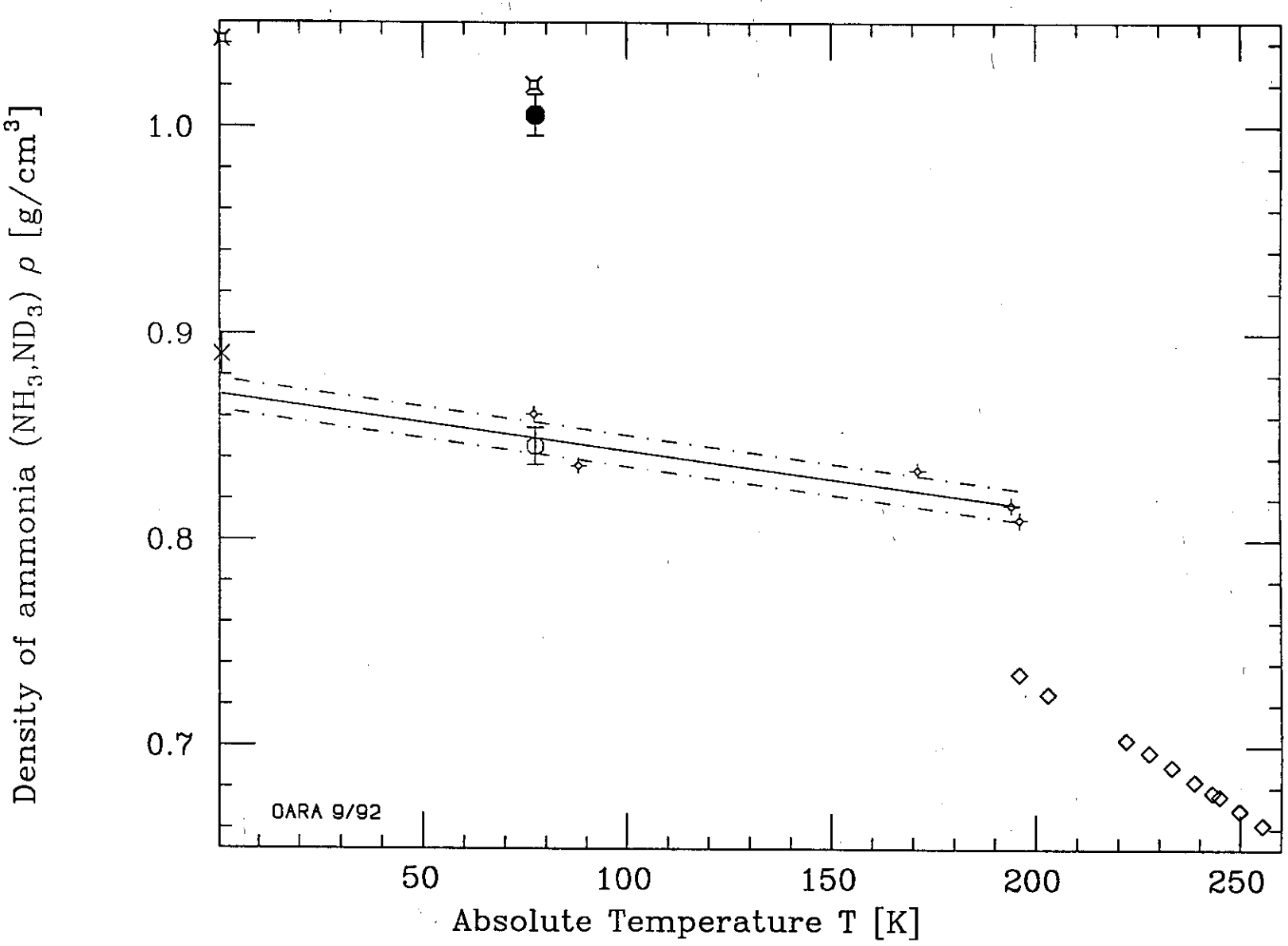
density of the liquid added, the calculation for the density of the ammonia is straightforward.

Results:

At the end of this report there is a graph of ammonia's density as a function of temperature. The results with the error bars are the averages of the values taken using our method. The measurements are in close agreement with other measurements made using other methods. Our values were 0.85 and 1.0 g/cm^3. To verify that our technique yielded accurate results, we measured several substances with known densities. We made measurements of ice, being careful to use distilled water frozen slowly. We also measured liquid nitrogen. The results showed conclusively that we could trust the measurements made by our technique.

Conclusion:

We were able to measure the density of frozen ammonia accurately without the trouble and expense of building an environment at -60C. Through the use of the computer to recoup our losses due to evaporation we were able to compensate and arrive at very accurate results.


Density of ammonia (NH_3,ND_3) ρ [g/cm^3]
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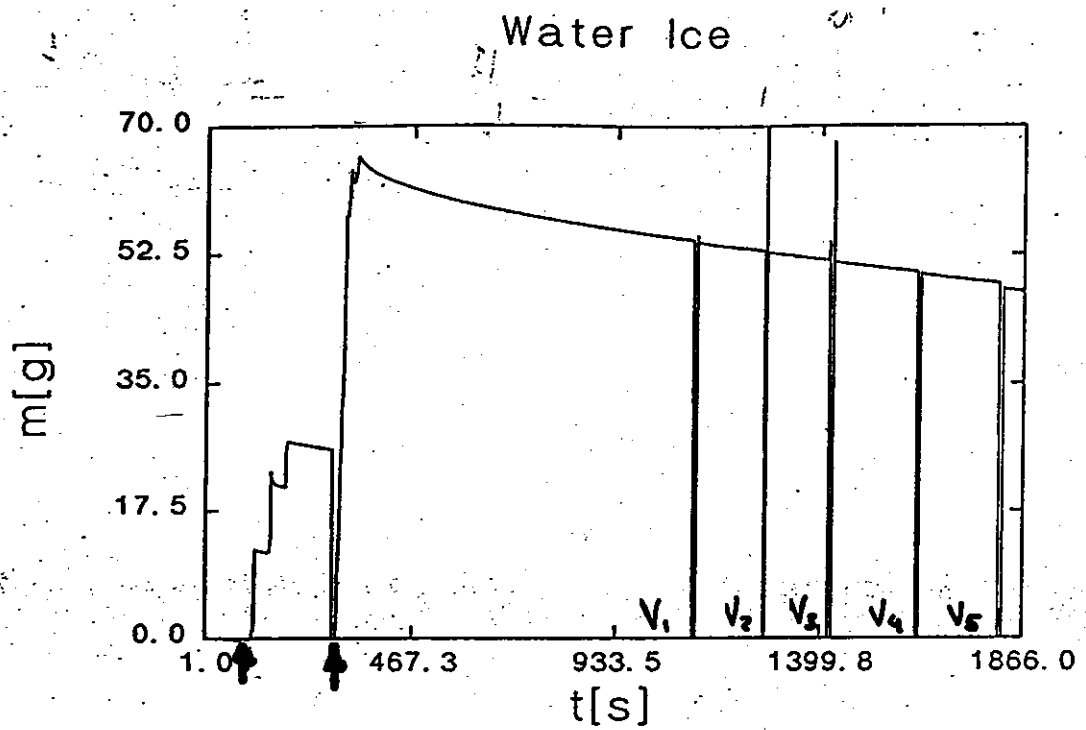
0.7
OARA 9/92
50
100
150
200
250
Absolute Temperature T [K]


Water Ice
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17.5
0.0
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1.0
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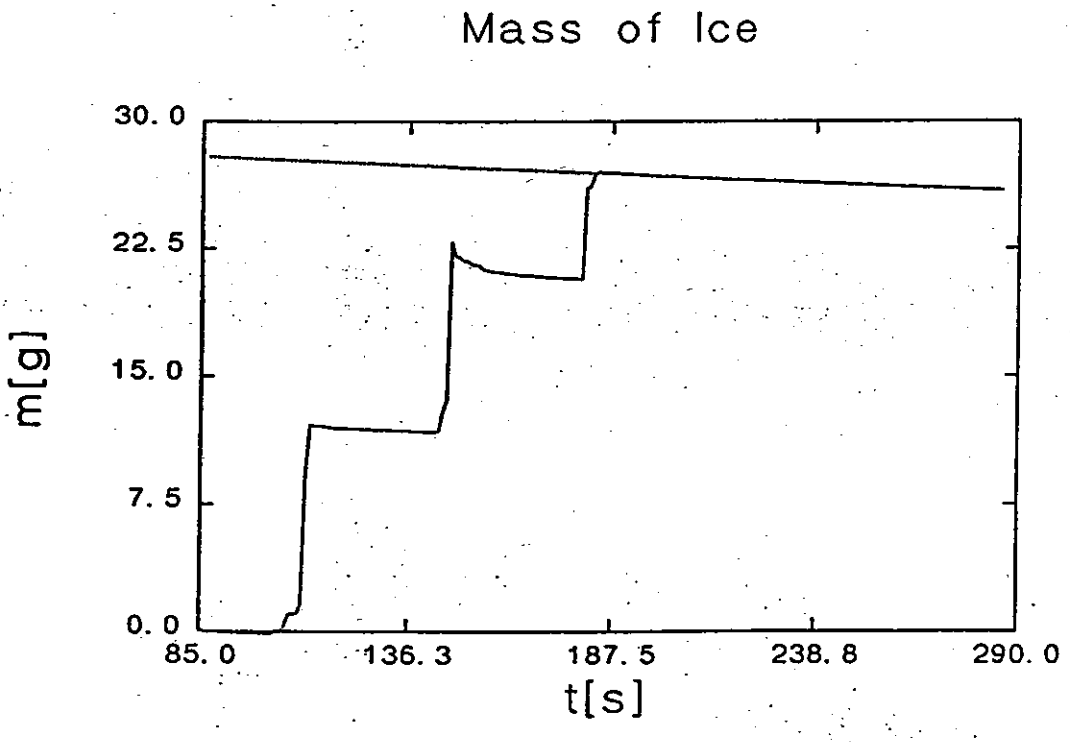
t[s]
V1
V2
V3
V4
V5

↑ = Taring.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7.5
0.0
m[g]
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136.3
187.5
238.8
290.0
t[s]