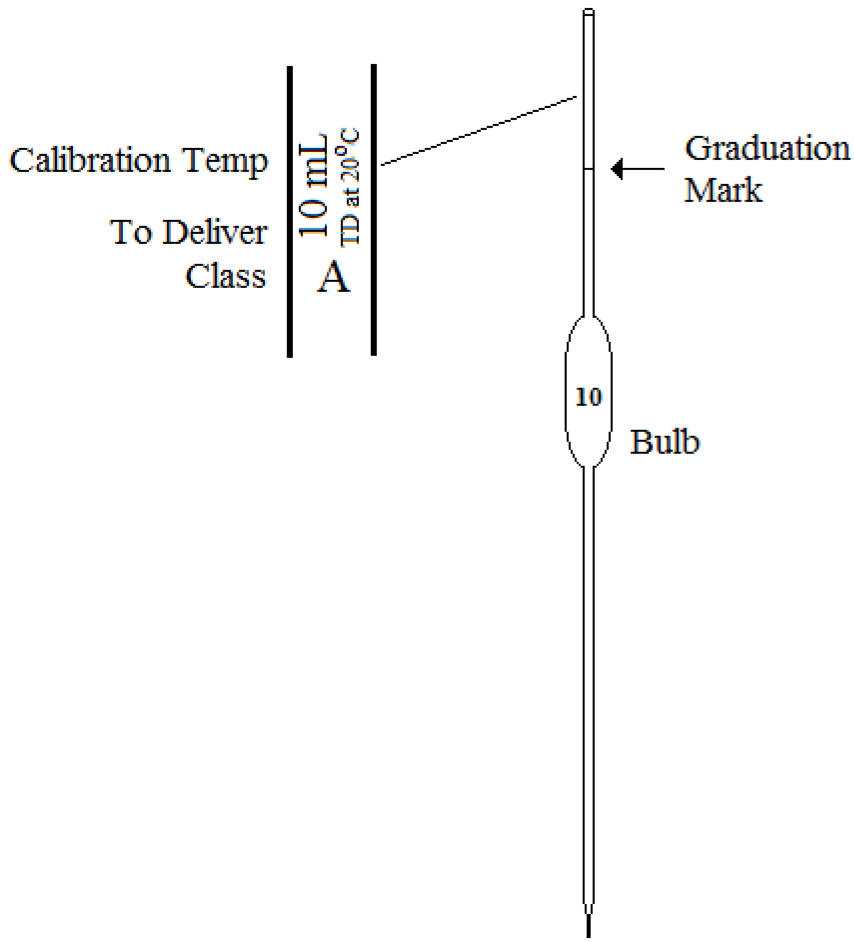
CHEM 109
Introduction to Chemistry
Fall 2011

# Laboratory Exercise:  The Precision of Volume Measurements

The accurate measurement of the volume of liquids tends to be difficult and chemists have a number of different types of devices for making this measurement; beakers, graduated cylinders, volumetric flasks, etc.  Each of these devices has a specific purpose and a distinct level of accuracy and precision associated with them.

A volumetric transfer pipette is a relatively accurate device for transferring a known volume of a liquid from one container to another.




**Volumetric Pipette**

Volumetric pipettes are graduated with a single mark to deliver a particular volume and typically come in sizes ranging from 0.1 mL to 100 mL.  The quality of the measurements obtained from these devices depends heavily on the care taken in calibrating and in using them.

In precise work it is never safe to assume that the volume delivered by or contained in any volumetric instrument is exactly the amount indicated by the calibration mark. Instead, recalibration is usually performed by weighing the amount of water delivered by or contained in the volumetric apparatus. The mass measurement is then converted into a volume measurement using the known density of Water. (See Appendix).

$$Volume = \frac{Mass}{Density}$$

This works well because mass measurements can be made much more accurately and precisely than can be volume measurements.

Example

Suppose we calibrate a 10 mL volumetric pipette by delivering Water at 24.0$^{o}$C into a previously weighed Erlenmeyer Flask. The new weight of the Flask plus the Water is then determined.

| | |
|---|---|
| Weight Flask + Water | 44.6999 g |
| Weight Flask Empty | <u>34.7182g</u> |
| | |
| Weight Water Delivered | 9.9817 g |

The volume of the pipette can then be determined:

$$\text{Volume} = \frac{9.9817\ g}{0.9972995\ g/mL} = 10.009\ \text{mL}$$

(Density obtained from the Appendix.)

We will make replicate measurements of the volume of Water delivered by a volumetric pipette to determine the precision with which we are able to use the device. We will then report the calibrated volume to the correct number of significant digits.

Many of the details concerning calibration of laboratory glassware can be found in the NIST publication “The Calibration of Small Volumetric Laboratory Glassware” by Josephine Lembeck; NBSIR 74-461. This publication can be found at:

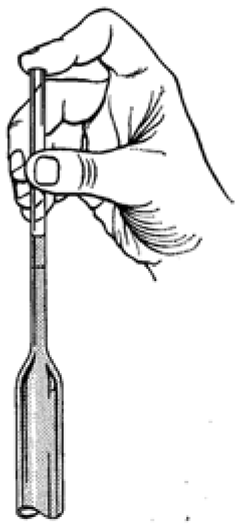
http://ts.nist.gov/MeasurementServices/Calibrations/upload/74-461.PDF

## Procedure

# *General Notes Concerning the Use of a Pipette*

Most Volumetric Pipettes are calibrated To-Deliver (TD); with a certain amount of the liquid remaining in the tip and as a film along the inner barrel after delivery of the liquid. The liquid in the tip should not be blown-out. Drainage rates from the pipette must be carefully controlled so as to leave a uniform and reproducible film along the inner glass surface.

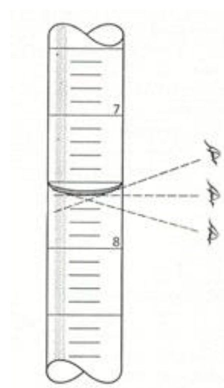
Once the pipet is cleaned and ready to use, make sure the outside of the tip is dry. Then rinse the pipet with the solution to be transferred. Insert the tip into the liquid to be used and draw enough of the liquid into the pipet to fill a smarall portion of the bulb. Hold the liquid in the bulb by placing your fore finger over the end of the stem.



Withdraw the pipette from the liquid and gently rotate it at an angle so as to wet all portions of the bulb. Drain out and discard the rinsing liquid. Repeat this once more.

To fill the pipette, insert it vertically in the liquid, with the tip near the bottom of the container. Apply suction to draw the liquid above the graduation mark. Quickly place a fore finger over the end of the stem. Withdraw the pipette from the liquid and use a dry paper to wipe off the stem. Now place the tip of the pipette against the container from which the liquid has been withdrawn and drain the excess liquid such that the meniscus is at the graduation mark. Make this determination with your eye level with the graduation mark so as to avoid a parallax error in the reading.

Move the pipette to the receiving container and allow the liquid to flow out (avoiding splashing) of the pipette freely. When most of the liquid has drained from the pipette, touch the tip to the wall of the container until the flow stops and for an additional count of 10.



# *Calibrating the Pippette*

1. First determine the temperature of the Room by measuring the temperature of a beaker of Water that has been allowed to equilibrate with the Room for several hours. This will be set-up for you in advance.

2. Obtain about 300 mL of Distilled Water in an 250 mL Beaker. This Water will be used to calibrate our pipettes.

3. Examine the pipette provided to you. Note:

   Graduated Volume
   Class
   Calibration Temperature
   Length of Delivery Tube
   Distance Between Bulb and Graduation Mark

4. Now, obtain a clean, dry 25or 50 mL Erlenmeyer flask with cork. Measure its mass using an **Analytical Balance**. (Always use the same analytical balance for repeat measurements.)

5. Use the pipette provided to you to deliver deionized Water whose temperature has been allowed to equilibrate with the Room Temperature into the flask.

6. Cork the flask and measure its mass.

7. Determine the volume of the pipette using the tabulated density of Water at the Room Temperature measured above.

8. Use a Kimwipe to dry the flask as best as is possible. Re-cork and weigh the flask again.

9. Repeat steps 4 – 8 until a total of five volume measurements have been made. Average these volumes to determine the "calibrated" volume for the pipette. Report the result to the correct number of significant digits. What is the level of the precision of your result?

# Appendix - Density of Water

| Temperature ($^oC$) | Density (g/mL) |
|---|---|
| 10 | 0.9997026 |
| 11 | 0.9996084 |
| 12 | 0.9995004 |
| 13 | 0.9993801 |
| 14 | 0.9992474 |
| 15 | 0.9991026 |
| 16 | 0.9989460 |
| 17 | 0.9987779 |
| 18 | 0.9985986 |
| 19 | 0.9984082 |
| 20 | 0.9982071 |
| 21 | 0.9979955 |
| 22 | 0.9977735 |
| 23 | 0.9975415 |
| 24 | 0.9972995 |
| 25 | 0.9970479 |
| 26 | 0.9967867 |
| 27 | 0.9965162 |
| 28 | 0.9962365 |
| 29 | 0.9959478 |
| 30 | 0.9956502 |

# Data Sheet

Water Temperature __________ $^{o}$C

Pipette Characteristics

Graduated Volume ______ mL

Class ______

Calibration Temp ______ $^{o}$C

Delivery Tube ______ mm

Bulb-to-Graduation ______ mm

Volume Measurements

| | Trial 1 | Trial 2 | Trial 3 | Trial 4 | Trial 5 | |
|---|---|---|---|---|---|---|
| Mass Filled | | | | | | g |
| Mass Empty | | | | | | g |
| Mass Wat | | | | | | g |

Name: ______________________ Date: ________________

Signature: ______________________

## Data Analysis

Show the Calculation of the Volume Delivered by the Pipette for Trial #1:

Tabulate the Results for all the Trials:

| | Trial 1 | Trial 2 | Trial 3 | Trial 4 | Trial 5 | |
|---|---|---|---|---|---|---|
| Volume | __________ | __________ | __________ | __________ | __________ | mL |

Report Your Final Results:

Av. Volume __________ mL (Report to the Correct Number of Significant Digits.)

Precision __________ mL

## Post Lab Questions:

1. Handling the stem of the pipette can lead to an error in the volume delivered by the pipette. Why is this and will this error be systematic or random? Explain. (Think temperature.)

2. In reading the room temperature, we have not worried about the calibration of the thermometer. Why is this?

3. Why does the pipette's graduation mark extended all the way around the barrel?

4. Does your pipette's length dimensions conform to the ASTM E969-02 standard? Why do you suppose these dimensions are important? (I am only asking you to speculate here.)

5. According the the ASTM E969-02 standard, what is the tolerance of your pipette? What is the tolerance of a measuring device and how does this differ from its precision?

6. Cleaning volumetric glassware is of critical importance. According to the NIST publication cited above, what solvents should be used to clean volumetric glassware?