

Chem 321 experiment 1

Environment, Water



CHEM 321 Experiment 1 Basics Review and Calibration of Volumetric

Glassware There are three types of containers used in lab to contain or deliver liquids: volumetric, ordinary, and disposable glassware. Volumetric glassware are containers that have been calibrated at a specific temperature to deliver or contain VERY PRECISE amounts of liquid. Examples of volumetric glassware that we will use include burets, pipets, and volumetric flasks. Ordinary glassware has less precise volume calibrations and are used whenever the volumes do not have to be measured as accurately. Examples include beakers, Erlenmeyer flasks, and graduated cylinders. Disposable glassware (or plastic ware) is used to transfer or hold liquids temporarily and may not contain any volume markings. Examples include medicine droppers and disposable pipets. In this experiment you will be exposed to a variety of important concepts related to quantitative experimentation, including the proper use of glassware, analytical balances, and statistics. You will calibrate a volumetric pipet or flask, that is, you will experimentally determine what volume a pipet or flask really delivers. You will also calibrate a beaker or Erlenmeyer flask and a 50 mL buret. You will use these calibrated values throughout the remainder of the quarter. Construct a table at the front of your laboratory notebook to record calibration values or tape in the calibration tables from Chapter 2 of Harris' 7th edition. How to read the level of liquid in glassware: The "meniscus" of a liquid refers to the curvature of the liquid surface in a narrow container. If the surface of the liquid is concave (as in water), the liquid level is measured using the bottom of the meniscus. If the surface is convex (upward curving) as in mercury, the liquid level is read from the top of the meniscus. It is important to position the eye at the

same level as the meniscus to avoid a parallax error. Sometimes it is easier to use an index card marked with a dark line or piece of black electrician's tape behind the glassware to contrast the line between the glassware markings and the liquid level. If the liquid level falls between the markings on graduated glassware, the student must estimate the last, uncertain digit in the recorded volume measurement. Example: some of our graduated cylinders have individual milliliter markings so a reading must be to the tenths of a milliliter (10.4 mL). Care of glassware: Glassware should be washed with a mild soap solution, rinsed with tap water, and then rinsed with distilled water. If beads of water stick to the inside walls, it should be cleaned again. If glassware is to be dried, allow it to drain or use paper towels. NEVER SLING GLASSWARE TO REMOVE WATER. NEVER TAP GLASSWARE AGAINST THE SIDE OF THE SINK. A buret or pipet should be rinsed with a small amount of the solution to be used to remove any water droplets from the glass walls. Burets: Burets must be read to the hundredths of a milliliter. Note that the liquid level markings begin at the top or open end. This is because the buret is designed to deliver liquids. All volumes are measured as the DIFFERENCE between an initial and final reading. Chem321 Exp. 1/Calib. of Glassware. Page 1 of 8 Pipet: The volumetric pipet is designed to deliver a single, fixed volume of liquid at a specific temperature. A graduated pipet is used in a similar fashion to the buret to deliver specific amounts of liquid. Remember to touch off the drop of liquid hanging outside the tip into the transferred liquid (it is part of the delivered volume) but DO NOT BLOW OUT ANY LIQUID THAT REMAINS INSIDE THE TIP OF THE PIPET. The pipet has been calibrated to contain this last drop of liquid. The

Analytical Balance The user of an analytical balance should know how to determine whether it is operating properly and how to make the simple adjustments that are occasionally necessary. A brief overview of analytical balances can be found in Section 2-3 of Harris. If you notice any inconsistencies in the performance of the balances, please notify either the instructor or the teaching assistant. It is important that an analytical balance be treated with the care due any precision instrument. We will be using electronic lab scales throughout the course. They are extremely sensitive and easily damaged. ALWAYS REMOVE ALL ITEMS FROM THE LAB SCALES AS SOON AS THEY INDICATE STABILIZATION. 1 to 3 seconds is usually enough time for the scales to stabilize. Never place a wet container on the scales. Never place or allow chemicals directly on the pan. Use massing papers or pans for chemicals. The TARE function clears the scales to zero. In this lab you will be finding mass by difference. Record the initial mass and then after adding or removing some component, mass again. Subtract the initial from the final mass to obtain the needed measurement. Some balances are equipped with a built-in compensator (taring mechanism) that enables the operator to set the balance to zero with an empty vessel on the pan. In this way, a sample can be weighed directly into a beaker or flask with only one weighing (excluding the zero reading) per sample. While this is the most efficient method, it is also less precise. In addition, weighing by difference enables hygroscopic or volatile samples to be weighed with little exposure to the atmosphere (the cap remains on the weighing bottle except for the time it takes to rapidly transfer some to the empty flask). Furthermore, weighing by difference prevents any spillage of chemicals to ever occur INSIDE the

balance. Potential sources of error in weighing with a constant-load balance include inaccurate weights, shifts in balance zero or sensitivity, and changes in the sample. Errors from weight changes in the sample include absorption of moisture (especially serious when a hygroscopic material is being weighed), volatilization, air currents from a hot sample or container, and static charge on containers. Use of the Balance: Never change balances in the middle of an experiment. Volumetric Measurements For making accurate measurements in analytical procedures, next in importance to the balance is volumetric equipment. In this handout, volumetric flasks, pipets, burets, and graduated cylinders are discussed. The experimental procedure includes gravimetric calibration of a pipet Chem321 Exp. 1/Calib. of Glassware. Page 2 of 8 to provide checks on the accuracy of the markings as well as on proper technique. The few minutes required to learn the correct use of this equipment will save time in the long run and give better results. For example, a typical experienced person using incorrect technique takes about 10 sec to read a buret and will have a standard deviation in the readings of 0.02 mL or more. A person of the same experience using correct technique takes about 6 sec and will have a standard deviation in the readings of 0.01 mL. Whenever you make chemical measurements, you need to understand the performance of your instruments and equipment. For example, although a pipet may be labeled "1 mL", this does not guarantee that it will deliver exactly 1mL. If you do not know the actual volume, you will be introducing an error into every operation that you perform with that pipet. Pipets are marked to deliver a known volume of water at a defined temperature, usually 20 or 25 degrees C. Volumetric Flasks Volumetric flasks are designed

to contain a specified volume of liquid. Rather than technique, the principal source of error is variation in temperature, which causes enough expansion or contraction of aqueous solutions to give errors on the order of 0.1% per 5 degrees C. Before use, a volumetric flask should be cleaned thoroughly. The solution to be diluted or the solid to be dissolved is then transferred to it with the aid of a funnel. Often it is more convenient for a solid to be dissolved in a beaker or a conical flask first, and then the resulting solution transferred quantitatively. In the most accurate work, the flask is filled to just below the mark and immersed in a water bath at the calibration temperature before final volume adjustment. Alternatively, the temperature of the solution may be taken and a correction made. In careful work droplets of solvent above the meniscus may be removed with a lintless towel after the meniscus has been adjusted but before mixing. Finally, the solution is mixed thoroughly by inverting, shaking, and turning upright at least 10 times.

Volumetric Pipets

Volumetric pipets are used to deliver precisely a single, definite volume of liquid. The tip must meet stringent requirements because drainage time is controlled by the diameter of the tip. The amount of liquid delivered depends on how a pipet is used; accuracies of 1 part per 1000 can be attained readily, provided the pipets are used in a reproducible manner. Keep in mind the following points. First clean the pipet so that water drains smoothly from the interior surface. Rinse the interior by drawing a portion of the liquid to be pipetted into the pipet with the aid of a suction bulb, and then tilt and turn the pipet until all of the inner surface has been wetted. Discard this portion of solution and repeat the operation. Then draw solution above the mark, wipe the tip and stem of the pipet carefully to remove external droplets,

touch the tip to the side of the beaker wall and allow the solution to drain until the bottom of the meniscus is even with the calibration mark. Touching the beaker wall draws liquid out of the pipet without leaving part of a drop hanging from the pipet when the liquid reaches the calibration mark.

Transfer the pipet to a receiving vessel and drain it by gravity while holding the tip against the wall of the vessel. The pipet should be held vertically, with the tip in contact with the container. Keep the tip in contact for about 5 sec after free solution flow has stopped and then remove it. The remaining liquid in the tip is supposed to be left there; do not blow out this portion. Chem321 Exp. 1/Calib. of Glassware. Page 3 of 8 Consult the instructor or a teaching assistant if you need refreshed on proper pipetting technique. The gloved index finger (not the thumb) is used to cover the end. If difficulty is experienced at first with a leaky gloved index finger, try moistening your gloved finger slightly; too much moisture, however, makes fine control of the outflow rate difficult. After a little practice pipetting will become both rapid and accurate. A rubber suction bulb is necessary for all pipetting. **DO NOT PIPET ANYTHING BY MOUTH.** Rinse the pipet thoroughly after use. Avoid drawing liquid into the bulb. If the bulb is contaminated accidentally, thoroughly rinse and dry it before re-use. Likewise, whenever a buret or pipet is used to deliver a measured volume of solution, the liquid in that item of glassware prior to measurement should have the same composition as the solution to be dispensed. As a result, always rinse your pipet with the solution to be measured prior to using it for the actual transfer. Burets Burets are designed to accurately deliver measurable volumes of liquid, particularly for titrations. A 50-mL buret, the most common size, has 0.1-mL graduations

along its length and can be read by interpolation to the nearest 0.01 mL. Parallax errors in reading are minimized by extension of every tenth graduation around the tube. For reproducible delivery, proper design of the tip is important. Changes affecting the orifice will affect reproducibility; a buret with a chipped or fire-polished tip should not be employed for accurate work. Drainage errors are usually minimized if the tip is constricted so that the meniscus falls at a rate not exceeding 0.5 cm/sec. The accuracy of graduation marks on volumetric burets depends on the diameter of the bore being uniform. The most convenient way to determine actual volumes is to weigh the amount of water delivered. Because graduations on a 50-mL buret are 0.1 mL apart, volumes between the marks must be estimated. In this estimation the width of the lines must be taken into account. The thickness of a line on a 50-mL buret is usually equivalent to about 0.02 mL and so takes up one fifth of the distance from one mark to the next. It is preferable to read the bottom of a meniscus. Parallax, another source of error in reading a buret, occurs if the eye is above or below the level of the meniscus. This error can be minimized by use of the encircling markings on the buret as guides to keep the eye level with the meniscus. The apparent position of the meniscus is significantly affected by the way it is illuminated. Lighting errors can be minimized by use of a white index card with a thick black line. The card is placed behind and against the buret so that the top of the black portion is flush with, or no more than a scale division below, the bottom edge of the meniscus. Alternately, placing a hand behind the buret makes it easier to see the meniscus. For some solutions, such as permanganate and iodine, the bottom of the meniscus may be difficult to

see; in such cases the top may be read. Before using a buret, clean it thoroughly, taking care not to scratch the interior surface. Rinse it well with distilled water; the buret is clean when water drains from the inside surface uniformly without the formation of droplets. Before use rinse the buret at least once with the titrant solution. After filling with titrant, make certain no air bubbles are present in the tip, especially just under the stopcock. Bring the solution level to slightly below the zero mark, remove the drop adhering to the tip by touching it to a clean piece of glass, and wait a few seconds for solution above the meniscus to drain before taking the initial reading. To provide effective control of the delivery rate when titrating, operate the stopcock with the left hand around the barrel (if right-handed). The other hand is free to swirl the titration flask. Calibration of Glassware Calibration of glassware is carried out by weighing the amount of water contained or delivered. Since the density, or weight per unit volume, of water changes about 0.03% per degree C at 25°C, the temperature of the water used in the calibration must be known. The actual volume delivered can be obtained from a table relating temperature to volume, which can be located in your textbook or on the internet. Although errors in buret and pipet markings by the manufacturer are usually small, they cannot be neglected in careful work. For example, if a 10-mL pipet actually delivers 9.990 mL, an error of one part per thousand will be introduced unless the true value is employed. 1) Make sure you understand the proper use of the analytical balances and volumetric pipets. Ask questions if you are unsure. Glassware preparation: Before you begin, make sure your glassware is clean. Water should run out smoothly, leaving a

thin film on the inside of the glass. It should not "bead" on the inside. 2) Place about 450 mL of deionized water in a 600 mL beaker with a thermometer. Allow the temperature of the water to equilibrate to room temperature. 50 mL Buret Calibration: Follow the write-up provided for you in your textbook. For Harris' Quantitative Chemical Analysis 7th edition, the procedure is located at the end of chapter 2, pg 38. Pipet Calibration: a) Weigh an Erlenmeyer flask to the nearest mg; record this value in your notebook. Note the specification given for the maximum weight allowance of the balance. Why is this important? b) Record the temperature of the water. Carefully fill the pipet with water up to the graduated mark and deliver the water to the flask in the appropriate manner. Record the mass. c) Repeat step (b) two-five additional times, using a second flask when the first flask becomes full. Depending on the initial weight of your Erlenmeyer flask, you should be able to make 2-3 weighings in each flask without discarding the water after each addition. d) Use the difference in mass between each set of two consecutive mass measurements to determine the mass of water delivered in each trial (thus the origin of the name "weighing by difference"). e) Calculate the true volume as in the example for a 10 mL volumetric pipet that follows. Duplicate calibrations should agree to within 0.01 mL. Use the average value for all subsequent measurements with this pipet. Chem321 Exp. 1/Calib. of Glassware. Page 5 of 8 Example for a 10-mL volumetric pipet: Temperature of Water: Weight of Flask and Water: Weight of Flask: Apparent Weight of Water Delivered: True Volume of Pipet: 26 oC 24. 688 g 14. 713 g 9. 975 g 10. 02 mL At 26 oC, 1 g of water occupies 1.0043 mL (Quantitative Chemical Analysis Table 2. 7). The true volume of 9.

975 g of water at 26 °C is $9.975 \text{ g} \times 1.0043 \text{ mL/g} = 10.02 \text{ mL}$ (Note: Confirm the proper number of significant figures.) Volumetric Flask Calibration: a) Note volume of volumetric flask calibrating. b) Weigh the clean and dried volumetric flask to the nearest milligram on an analytical balance. c) Fill the flask with distilled water at room temperature to just below the mark. d) Carefully add water with a disposable pipet until the bottom of the meniscus coincides with the calibration line. Remove droplets of water present above the line with a lintless towel or a strip of filter paper. e) Reweigh the flask. Immediately after weighing, measure and record the temperature of the water in the flask. Calculate the true volume of the flask in the same way as for the volumetric pipet. Obtain at least three measurements by emptying the flask and drying it between measurements. Air drying is best if time permits, DO NOT use the house air lines. If you are careful in keeping track of your glassware you can alternate glassware weighings to help in the drying process. Beaker Calibration: a) Place a clean, dry beaker on the lab scales and record the mass of the beaker in your lab notebook. b) Remove the beaker from the balance and add some of the tap water into it. Using the graduations on the beaker, read and record the volume of water. c) Place the beaker on the scales. Calculate (by subtracting the mass of the beaker) and record the mass of the water in the beaker. d) TARE the scales and repeat for trials 2 and 3 (up the five trials if you choose). (Add more water, read the amount, mass). Chem321 Exp. 1/Calib. of Glassware. Page 6 of 8 Graduated Cylinder Calibration: a) Place a clean, dry graduated cylinder on the lab scales. Record mass. Remove from lab scales. b) Pour some tap water into the graduated cylinder. Remember to

remove any water bubbles from the liquid first and then remove any droplets from the inside or outside of the cylinder. Using the graduations on the cylinder, read the volume as precisely as possible and record. c) Place the cylinder on the scales and calculate/record the mass of the water. d) Repeat for trials 2 & 3 (up the five trials if you choose). Remember that you need not clean and dry the cylinder between each trial. Calculations for Volumetric Equipment Calibration: 1) Enter the calculated masses and volumes in your laboratory notebook. 2) Calculate: - - - - the mean (average) volume the standard deviation the relative standard deviation (%RSD) the 95% confidence interval the % relative error (assume the theoretical volume is exactly 25 mL for the pipet and 100 mL for the volumetric flask) 3) Construct a reference table of this data and place it on the 2nd or 3rd page of your lab notebook, leaving space for your Table of Contents. You will use the mean and standard deviation of your calibrated glassware throughout the rest of the quarter. Things to consider through-out the experiment and to include in your formal lab report: 1) Comment on whether or not the correction volume is within the error allowed for your volumetric glassware. 2) Comment on the difference between volumetric and ordinary glassware. 3) Why is it important to do a temperature conversion for the water that you used while calibrating your glassware? What were the corrections that you took into account? 4) Most volumetric glassware is calibrated at what temperature? Chem321 Exp. 1/Calib. of Glassware. Page 7 of 8 5) What sort of errors do you expect from the analytical balance measurements? What precision can you expect from the analytical balance? What if you forgot to tare the balance? 6) What is the best way to read a buret? 7) What do the letters T. C. and T. D. that are

found on various types of volumetric glassware signify? Chem321 Exp.
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