

## **Calibration of Volumetric Ware and Weighing**

(Adapted from Text: Section 35I, pages 806-808)

### **Calibration of Volumetric Ware**

**Preparation.** Read Section 35H - The Measurement of Volume and Section 35I - The Calibration of Volumetric Ware.

**Note.** Fill a 500 mL beaker with water well in advance and note its temperature at frequent intervals. When it has reached thermal equilibrium with its surroundings, it can be used for the calibrations to be performed.

**Cleaning.** All glassware should be washed with (warm) detergent before use. Clean glass surfaces leave a uniform film of water as they drain. Dirt or oil causes breaks in this film; breaks are a certain indication of an unclean surface. Ordinarily, calibrated glass equipment does not need to be dried. However, the volumetric flask must be thoroughly drained and *air dried* before it can be calibrated.

As a general rule, calibrated glass equipment should *not* be heated.

**Calibration of a Volumetric Pipet.** Determine the weight of a stoppered conical weighing bottle to the nearest milligram. Fill your 25 mL pipet with temperature-equilibrated water and transfer it to the receiver. Weigh the receiver and its contents, and calculate the weight of water delivered from the difference in these weights. Calculate the volume delivered with the aid of Table 35-3 in the text. Note that the numbers in this table are not the density of water but the volume in mL per gram of water (corrected for buoyancy and temperature effects).

**Calibration of a Volumetric Flask.** Use the top-loading balance provided for this experiment. The analytical balances do not have the capacity to weigh a water-filled volumetric flask. Weigh the clean, dry 100 mL volumetric flask to the nearest milligram. Then fill to the mark with equilibrated water and reweigh. A medicine dropper is useful in making final adjustments to the liquid level. Calculate the volume contained with the aid of Table 35-3. Use this 100 mL of water for the next procedure.

**The Aliquot Method.** A calibrated volumetric flask and pipet provide an excellent method for partitioning a sample into identical aliquots. Empty and reweigh the stoppered weighing bottle. Carefully transfer a 25 mL aliquot from the 100 mL volumetric flask to the weighing bottle and determine its weight. Determine the weight of two more aliquots in the same way. Convert the weights to volumes using Table 35-3. These data provide three additional values for the pipet calibration. This technique will be used in several of the experiments. Each aliquot contains one-fourth of the contents of the volumetric flask. If their weights agree within  $\pm 20$  mg you are doing

OK. Check with your instructor if the volume of your volumetric flask or pipet seem to be quite different from their nominal values.

***Calibration of a Buret.*** Fill the buret above the 0.00 mL mark with temperature-equilibrated water. Ensure that there are no bubbles trapped in the tip and that it drains without leaving drops on the walls. Lower the liquid level to bring the bottom of the meniscus to the 0.00 mL mark or just below it. Touch the tip to a beaker to remove any adhering drop of water. Record the initial volume reading, estimating to the nearest 0.01 mL. Let the buret sit for about five minutes while you weigh the weighing bottle. If the volume changes during this time, there may be a leak and the stopcock needs tightening. Slowly (at about 10 mL per minute) deliver about 20 mL of water to the weighing flask. Reweigh the flask to determine the mass of water delivered and record the buret reading again. Now transfer an additional 10 mL to the weighing bottle while draining the buret to the 30 mL mark. Reweigh the bottle and read the buret level. Repeat the procedure at 40 mL and 50 mL. Repeat the whole procedure. Convert the weights of water to volumes and compare with the buret readings. Prepare a table of the differences between these two values as a function of volume delivered (agreement within  $\pm 0.05$  mL is reasonable). At this point in the course, this "calibration curve" may be more indicative of your technique than a reliable measure of the buret accuracy.

## Weighing by Difference

In this experiment we weigh by difference, and then check for the accuracy of the transfer by weighing the receiving bottle. Nearly every analysis you do requires such expertise. Once the technique is mastered, it will no longer be necessary to weigh the container to which the transfer was made. In practice, we do not transfer from one weighing bottle to another, but rather transfer the sample to beakers, flasks, etc. (which typically are too heavy to weigh on an analytical balance).

**Preparation:** Read Sections 35A, B, C, D, & E, pp 778- 790.

### **Procedure:**

- Clean and thoroughly dry three weighing bottles and their stoppers.
- Two rules: no reagent bottles in the balance room and never leave spilled reagents in or around a balance.
- Weigh approximately three grams of sodium chloride using the beam balance and a weighing boat. Transfer the salt to one weighing bottle (#1). Stopper and weigh it using the analytical balance (0.1 mg precision).
- Weigh the other two weighing bottles (#2 and #3).
- Carefully transfer a sample of about 0.5 gram from #1 to #2, avoiding loss. This takes practice. If you spill any, try again. Do this operation over a sheet of paper to assist in containing any spilled material.
- Determine the weights of #1 and #2. The weight gain of #2 should equal the weight loss of #1 within a few tenths of a milligram.
- Repeat the process, transferring about 0.5 g from #1 to #3 and weighing each bottle.
- Repeat the process, transferring about 0.4 g from #2 to #3 and weighing each bottle.
- Pour the contents of #2 and #3 back into #1. The NaCl can now be oven-dried in preparation for the determination of chloride experiment.

## Report

All of the data should be recorded in your notebook following the procedures in Section 35J, p 808 in your text. This should be available for inspection, if requested. Prepare a summary of your results using a spreadsheet. An example is shown below. For the calibration of the pipet, this includes a table listing the weights of water delivered and their calculated volumes (using Table 35-3) together with the mean volume delivered and its standard deviation. Are there any outliers (Section 4C, pp 57-59)? For the weighing by difference exercises prepare a table showing the weight loss/gain after each transfer. Assuming the receiving bottle gives the true value, report the absolute error and the relative error (expressed as a percent and in parts per thousand) for each transfer. The results for the calibration of the volumetric flask and the buret are not included in the example but should be included in your report.