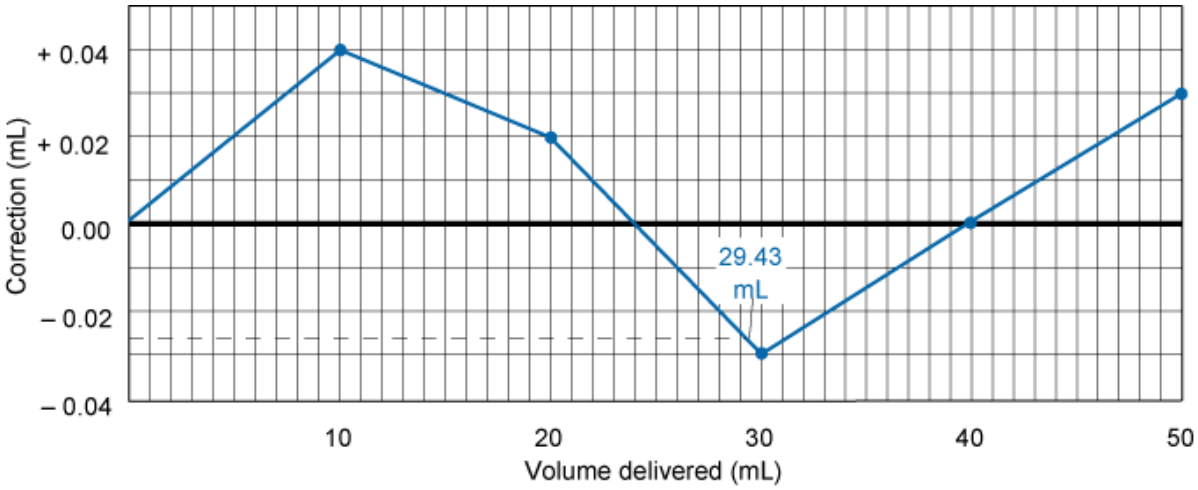
1. Calibration of Volumetric Glassware

Green Profile
See Section 0

An important trait of a good analyst is the ability to extract the best possible data from his or her equipment. For this purpose, it is desirable to calibrate your own volumetric glassware (burets, pipets, flasks, etc.) to measure the exact volumes delivered or contained. This experiment also promotes improved technique in handling volumetric glassware.

Calibrating a 50-mL Buret

This procedure tells how to construct a calibration graph such as the one below to convert the measured volume delivered by a buret to the true volume delivered at 20°C.



1. Fill the buret with distilled water and force any air bubbles out the tip. See whether the buret drains without leaving drops on its walls. If drops are left, clean the buret with soap and water or soak it with cleaning solution.[1] Adjust the meniscus to be at or slightly below 0.00 mL (0.00 to 0.30 mL), and touch the buret tip to a beaker to remove the suspended drop of water. Allow the buret to stand for 5 min while you weigh a 125-mL flask fitted with a rubber stopper. (Hold the flask with a tissue or paper towel, not with your hands, to

1. Prepare cleaning solution by dissolving 36 g of ammonium peroxydisulfate, $(NH_4)_2S_2O_8$, in a *loosely stoppered* 2.2-L ("one gallon") bottle of 98 wt% sulfuric acid. Add ammonium peroxydisulfate every few weeks to maintain the oxidizing strength. EOSULF is an alternative cleaning solution for removing proteins and other residues from glassware in a biochemistry lab. EOSULF contains the metal binder EDTA and a sulfonate detergent. It can be safely poured down the drain. [P. L. Manske, T. M. Stimpfel, and E. L. Gershey, *J. Chem. Ed*. **1990**, *67*, A280.]

prevent fingerprint residue from changing its mass.) If the level of the liquid in the buret has changed, tighten the stopcock and repeat the procedure. Record the level of the liquid.

2. Drain approximately 10 mL of water at a rate < 20 mL/min into the weighed flask, and cap it tightly to prevent evaporation. Allow about 30 s for the film of liquid on the walls to descend before you read the buret. Estimate all readings to the nearest 0.01 mL. Weigh the flask again to determine the mass of water delivered.

3. Now drain the buret from 10 to 20 mL, and measure the mass of water delivered. Repeat the procedure for 30, 40, and 50 mL. Then do the entire procedure (10, 20, 30, 40, 50 mL) a second time.

4. Use the table of water density at the end of this experiment to convert the mass of water into the volume delivered. Repeat any set of duplicate buret corrections that do not agree to within 0.04 mL. Prepare a calibration graph like the one on p. 1, showing the volume correction at each 10-mL interval.

EXAMPLE Buret Calibration

When draining the buret at 24˚C, you observe the following values:

Final reading	10.01	10.08 mL
Initial reading	0.03	0.04
Difference	9.98	10.04 mL
Mass	9.984	10.056 g
Actual volume delivered	10.02	10.09 mL
Correction	+0.04	+0.05 mL
Average correction	+0.045 mL	

To calculate the actual volume delivered when 9.984 g of water are delivered at 24˚C, look in the table of water density on p.4 at the column headed "Corrected to 20^{o}C." In the row for 24oC, you will find that 1.0000 g of water occupies 1.0038 mL. Therefore, 9.984 g occupies (9.984 g)(1.0038 mL/g) = 10.02 mL. The average correction for both sets of data is +0.045 mL.

To obtain the correction for a volume greater than 10 mL, add successive masses of water collected in the flask. Suppose that the following masses were measured:

Volume interval (mL)	Mass delivered (g)
0.03–10.01	9.984
10.01–19.90	9.835
19.90–30.06	10.071
Sum 30.03 mL	29.890 g

The total volume of water delivered is (29.890 g)(1.0038 mL/g) = 30.00 mL. Because the indicated volume is 30.03 mL, the buret correction at 30 mL is –0.03 mL.

What does this mean? Suppose that the calibration graph shown on p. 1 applies to your buret. If you begin a titration at 0.04 mL and end at 29.00 mL, you would deliver 28.96 mL if the buret were perfect. The calibration graph tells you that the buret delivers 0.03 mL less than the indicated amount; so only 28.93 mL were actually delivered. To use the calibration curve, either begin all titrations near 0.00 mL or correct both the initial and the final readings. Use the calibration curve whenever you use your buret.

Calibrating Other Glassware

Other volumetric glassware can also be calibrated by measuring the mass of water they contain or deliver. Glass transfer pipets and plastic micropipets can be calibrated by weighing the water delivered from them. A volumetric flask can be calibrated by weighing it empty and then weighing it filled to the mark with distilled water. Perform each procedure at least twice. Compare your results with the tolerances listed in tables in Chapter 2 of the textbook.

Density of water

Temperature (°C)	Density (g/mL)	Volume of 1 g of water (mL)	
		At temperature shown[a]	Corrected to 20°C[b]
10	0.999 702 6	1.001 4	1.001 5
11	0.999 608 4	1.001 5	1.001 6
12	0.999 500 4	1.001 6	1.001 7
13	0.999 380 1	1.001 7	1.001 8
14	0.999 247 4	1.001 8	1.001 9
15	0.999 102 6	1.002 0	1.002 0
16	0.998 946 0	1.002 1	1.002 1
17	0.998 777 9	1.002 3	1.002 3
18	0.998 598 6	1.002 5	1.002 5
19	0.998 408 2	1.002 7	1.002 7
20	0.998 207 1	1.002 9	1.002 9
21	0.997 995 5	1.003 1	1.003 1
22	0.997 773 5	1.003 3	1.003 3
23	0.997 541 5	1.003 5	1.003 5
24	0.997 299 5	1.003 8	1.003 8
25	0.997 047 9	1.004 0	1.004 0
26	0.996 786 7	1.004 3	1.004 2
27	0.996 516 2	1.004 6	1.004 5
28	0.996 236 5	1.004 8	1.004 7
29	0.995 947 8	1.005 1	1.005 0
30	0.995 650 2	1.005 4	1.005 3

a. Corrected for buoyancy with Equation 2-1 in the textbook.

b. Corrected for buoyancy and expansion of borosilicate glass (0.001 0% K^{-1})