Chem 321 Lecture 3 - Analysis, Measurements and Tools
9/3/13

Student Learning Objectives

Mass Measurements

Two of the most important types of measurements you will make in the laboratory involve mass and volume. Masses of small objects (≤200 g) can be very precisely determined using an analytical balance. Such a device is capable of weighing samples to the nearest 0.1 mg. A top-loading balance is used to weigh larger objects or to obtain approximate masses. Both types of balances are easy to use and operate on principles associated with the design shown in the figure below.

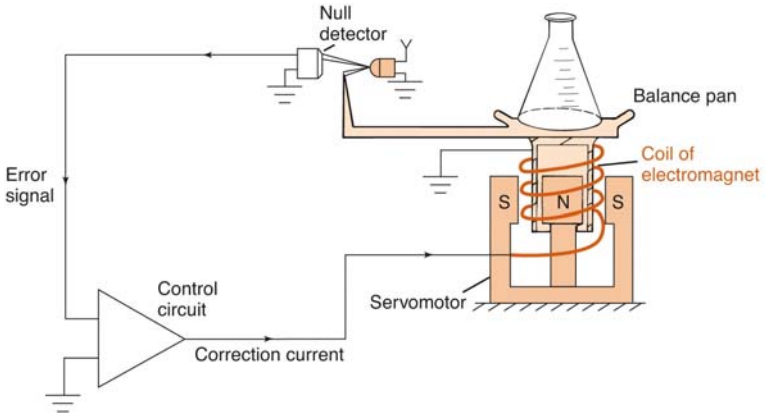

Figure 1.1 Basic circuit design of an electronic balance

The balance pan is associated with a detector arm and a coil of wire through which a current can flow. Initially the balance is zeroed with no sample on the pan. This fixes the location of the balance arm with respect to the detector. Then a calibrated internal mass (200 g in our analytical balances) is placed on the balance pan. This causes the pan to move downward. This movement is sensed by the detector and a signal is sent to the control circuit to send current through the coil of wire attached to the pan. Current flowing through this coil creates a magnetic field that interacts with the magnet surrounding the coil to cause the pan to move upward (more current results in more movement of the pan). The current is increased until the pan is raised to its original position with no sample on the pan. At this stage, the balance has two (mass,current) data points with which to electronically construct a linear calibration curve (imagine a plot of current vs. mass on pan). The standard mass is then removed. When another object is placed on the pan, the current needed to restore the pan to its original position is noted and the mass of the object is determined by interpolation on this calibration plot. The mass value is read off a digital display.

Check for Understanding 1.2 **Solutions**

1. What is the relative error associated with a mass measurement of 0.4887 g?

2. What are the *x*-coordinates for the two data points used to calibrate our analytical balances?

Despite their ease of use, analytical balances can be a source of significant error unless proper techniques are followed. Important sources of weighing errors include temperature effects, sample contamination and buoyancy effects.

Temperature Effects

Mass measurements may be in error when the temperature of the sample is different than the ambient air temperature. Samples that are too cool (e.g., right out of the refrigerator) will cause moisture in the air to condense of the sample, thus causing the reading to slowly increase. Samples that are too hot (e.g., right out of the oven) will create air currents that result in fluctuating, and usually decreasing, readings. When weighing samples that have been heated in the oven, wait a minimum of 30 minutes for the sample to come to room temperature. During this time the sample must be stored in a dry environment to prevent it from picking up moisture from the air. Your **desiccator** provides a dry atmosphere for storage of samples while cooling. An ordinary desiccator (Fig. 1.2) is a thick-walled glass container with a ground glass surface for the lid. A small amount of grease on the ground glass surface serves to make an airtight seal. A drying agent called a **desiccant** is placed in the bottom of the desiccator to maintain a dry environment. The lid should be removed only when moving samples in and out of the dessicator. **Place only materials that have been thoroughly dried in your desiccator, never wet or undried materials.**

Figure 1.2 Ordinary desiccator

Sample Contamination

When weighing samples to the nearest 0.1 mg, direct handling of the samples should be avoided. Oils and perspiration from your fingers will affect the mass of your sample noticeably. Use tongs or a strip of Kimwipe to handle glassware that is being weighed. In a similar way, sample contamination can result from putting a sample on a soiled lab surface. This includes a dirty balance pan. Place samples on a clean Kimwipe if they are to be set on the bench before weighing. The following laboratory practices will minimize sample contamination and help maintain the balances in the lab so that they function properly and provide reliable mass measurements for all users. These practices should be followed at all times.

Proper Sample Handling Practices

- Clean up all spills around the balances immediately.
- Never weigh reagents directly on the balance pan. Always use a small glass or plastic container to hold reagents while weighing. Do not use weighing paper because it is difficult to contain loose reagent samples on the paper.
- **Never put any object (e.g., a spatula or pipet) into a reagent bottle to extract a sample.** Tap out or pour the material into another container before taking an aliquot.
- Never return unused reagents to their original container.

Buoyancy Effects

All objects in air are buoyed up by a force dependent upon the amount of air they displace (Archimedes' principle). Consequently, the magnitude of this effect depends on the density of the object. Recall how the analytical balance is calibrated using a 200-g internal mass, which is also subject to air buoyancy. If a 200-g sample with a different density than that of the internal mass is placed on the balance pan, it will be buoyed up by a different amount than that of the internal mass because it displaces a different volume of air. Hence, the balance pan will not be displaced by the same amount as with the internal mass and the apparent mass of the sample (that given on the digital display) will not be 200 g. Thus, any sample that has a density different than

that of the calibration mass (density = 8.0 g/cm^3) will have an apparent mass that is in error due to buoyancy. The relevant question is: How big is this error?.

The magnitude of this effect can be calculated using the equation below.

$$m_t = m_a + m_a\left(\frac{d_{air}}{d_{obj}} - \frac{d_{air}}{d_{int'l\,mass}}\right)$$

where m_t = true mass of the object (g)
m_a = apparent mass of the object (g)
d_{air} = density of air (= 0.0012 g/cm^3 for dry air at 25 °C)
d_{obj} = density of object (g/cm^3)
$d_{int'l\ mass}$ = density of internal calibration mass (= 8.0 g/cm^3)

Substituting and rearranging this equation gives:

$$\frac{m_t}{m_a} = 1 + 0.0012\left(\frac{1}{d_{obj}} - \frac{1}{8.0}\right)$$

If the object density equals 8.0 g/cm^3, the quantity in parentheses is zero, $m_t / m_a = 1$ and there is no buoyancy error (the reading is the correct mass).

However, if the object is water (d = 1.0 g/cm^3) or glass (d = 2.7 g/cm^3), there will be a buoyancy effect and the true mass of the object will not equal its apparent mass.

d_{obj} (g/cm^3)	m_t / m_a
8.0	1.0000
1.0	1.001
2.7	1.0003

Check for Understanding 1.3 **Solutions**

1. Calculate the ppth buoyancy error in the mass reading for an object having a density of:

   a) 1.0 g/cm^3 b) 2.7 g/cm^3

Since we are generally concerned with ppth levels of error in our lab work, buoyancy errors will not be significant when typical solids are being weighed. However, this is not the case for the water samples weighed during the calibration of your volumetric glassware. Note that the buoyancy error depends on the ratio of the object density and the internal mass density, not the difference. The more this ratio of densities (calculated as a quantity > 1) differs from unity, the bigger the buoyancy error.

Volume Measurements

Careful volume measurements will be done using Class A (analytical grade) volumetric glassware (burets, pipets and volumetric flasks). Generally the uncertainties associated with volumes measured using these tools are on the order of ppth relative error. The tolerances for Class A volumetric flasks and Class A glass transfer pipets are given in the table below.

Tolerances for Class A volumetric flasks		Tolerances for Class A transfer pipets	
Flask capacity (mL)	Tolerance (mL)	Volume (mL)	Tolerance (mL)
1	±0.02	0.5	±0.006
2	±0.02	1	±0.006
5	±0.02	2	±0.006
10	±0.02	3	±0.01
25	±0.03	4	±0.01
50	±0.05	5	±0.01
100	±0.08	10	±0.02
200	±0.10	15	±0.03
250	±0.12	20	±0.03
500	±0.20	25	±0.03
1000	±0.30	50	±0.05
2000	±0.50	100	±0.08

Check for Understanding 1.4 **Solution**

1. What is the ppth relative error associated with a 2.0-mL volume delivered using a Class A transfer pipet?

Guidelines for Using Volumetric Glassware

- Make sure the equipment is clean. Water will not bead up on the inside walls if it is clean. If cleaning is required, soak the inside surfaces in a cleaning solution consisting of 0.07 M $(NH_4)_2S_2O_8$ in concentrated H_2SO_4.

- Never store alkaline solutions in volumetric glassware. The basic solution attacks glass.

- Never directly heat volumetric glassware. To promote dissolution of solutes when preparing solutions in a volumetric flask, put the flask in a beaker of hot water.

- When preparing an aqueous solution in a volumetric flask, first dissolve or dilute the sample in a small volume of water so that the contents can be thoroughly mixed by swirling. Continue to swirl the solution as more water is added. When the solution is finally diluted to the marked, secure the stopper and invert the flask at least a dozen times with swirling/shaking.

- Glass transfer pipets are calibrated “to deliver” (TD). That is, the liquid that fills the pipet to the calibration line is allowed to drain from the pipet, with the last hanging drop removed. Any residual liquid is not blown or shaken from the pipet.

- Make all buret readings to the nearest 0.01 mL. Note that the thickness of the buret calibration lines is about 0.02 mL. Establish a convention to allow for this, such as when the meniscus is exactly at the top of a line the hundreths place is zero. See examples of buret readings in Figure 1.3.

- Before dispensing liquid in a buret, ensure that there is no air bubble trapped beneath the stopcock. If there is, fully open the stopcock to allow liquid to expel the bubble.

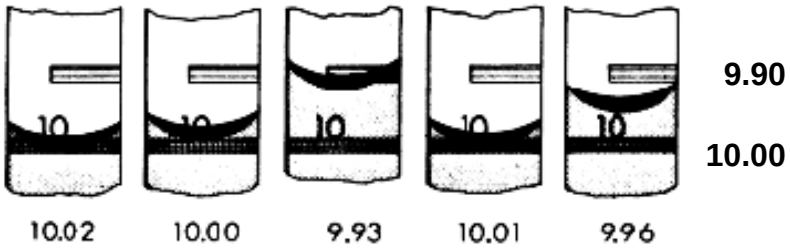

Figure 1.3 Enlarged sections of a 50-mL buret showing the meniscus at several positions

Mechanical Pipets

Another convenient tool for dispensing liquids, especially repetitively, is the autopipet. This is an attractive alternative to the transfer pipet because it employs disposable plastic tips that eliminate the need to clean the pipet when dispensing different liquids. However, the tolerance for autopipets is generally larger than for Class A glass transfer pipets. The fixed volume autopipet you will be using in lab has a tolerance at 1.0 mL of 1.000 ± 0.008 mL.

Although autopipets are very easy to use, sufficient care must be exercised when making deliveries or large errors can be introduced. In particular, it is important to check that all of the liquid is expelled from the tip when making a delivery. These pipets are “to contain” (TC) instruments which means that all of the liquid drawn into the tip must be expelled. A good practice is to rinse the tip with the solution into which you pipet to ensure that all of the sample is removed from the tip. However, this tip should not then be used to dispense more sample. For our Rainin autopipets it is recommended that you first fill and expel two volumes of the liquid you are pipeting before making your final delivery.

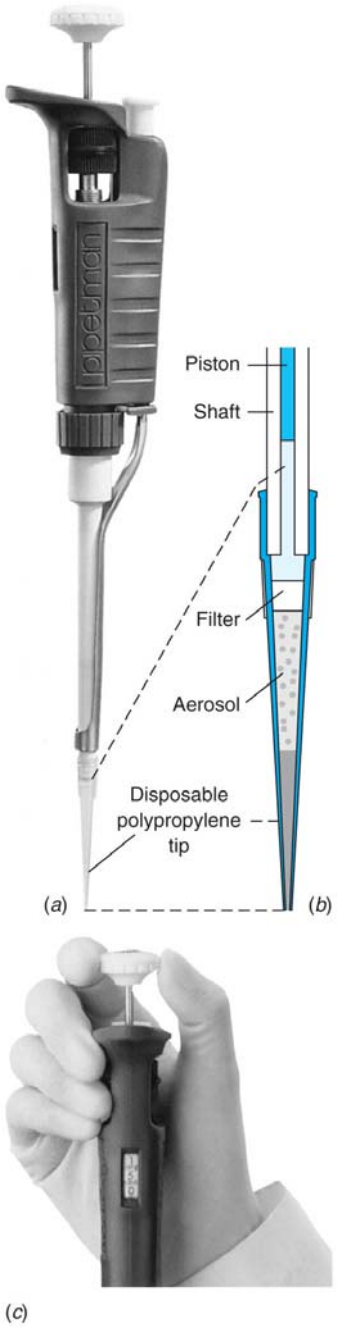

Figure 1.3 (a) Autopipet with disposable tip
(b) Enlarged view of disposable plastic tip
(c) Adjustable pipet volume control

Buret and Pipet Calibrations

Your initial work in the lab will be to calibrate your buret, 2-mL transfer pipet and the autopipet you are assigned. These calibrations may seem tedious, but they are very important because they will allow you to correct for ppth errors associated with the

use of each item in various experiments. The calibration process involves the weighing of water samples delivered by either the buret or pipet. The mass of the water samples weighed during the calibration of the pipets and buret will be corrected for buoyancy using the mass-to-volume conversion factors in the table below.

Table 2-7 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.

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

The factors in column 4 are used for the glass transfer pipet and buret because they include a correction for glass expansion. The factors in column 3 are used for the autopipet calculations. In each case, the mass of the dispensed water sample is multiplied by the conversion factor (units = mL/g) associated with the your measured water temperature to convert it to the actual delivered volume.

Exercises for Analysis, Measurements and Tools