

QUANTITATIVE DETERMINATION OF METAL IN A SOLID

	DETAILS	DUE DATE
DURATION OF LAB:	August 25-28, 2003	September 2, 2003
READING ASSIGNMENT:	pp 1-29, pp. 45-58*	
PROBLEM ASSIGNMENT:	2-8, 3-1, 3-5, 3-14, 27-16	August 26, 2003
ADDITIONAL ASSIGNMENTS:	Read: J.Environ.Qual-1989-18-374	

*All page number in reference to 6th Ed. Quantitative Analysis, D.C. Harris

Background

Quantitative descriptions of samples or substances are often made through mass, mass ratios, or percent weight by element or compound (p 19). For example, fertilizers are often classified and used on the basis of weigh percent of nitrogen (percent nitrogen weight to weight, or %N wt/wt, see <http://www.gov.on.ca/OMAFRA/english/crops/facts/90-201.htm>). Another example is fat content in ice cream (http://www.formatinternational.com/examples/ice_cream_manufacturing.htm). In this laboratory, you will be given a sample containing metal and a soluble solid. Your interest is in determining the percent metal in the sample using gravimetric methods.

Percent metal in your sample can be found using the formula:

$$\text{Percent Metal} = \frac{\text{Grams of Metal}}{\text{Grams of Sample}} \times 100$$

A second facet of this laboratory is the calculation of error in your measurement. All measurements contain indeterminate or random error. While this error is often incorporated into a standard deviation when multiple (or replicate) measurements are made, error can be calculated also for a single determination through propagation of error formulas (pp. 51-58).

The next aspects in this experiment are that of bringing a sample to constant weight and of weight by difference. In the procedure of constant weight, moisture in your sample or glassware is brought to a constant and ideally correct value through repeated heating, cooling, and weighing of your sample (pp. 36-37). Weight by difference is the determination of the weight of a substance by indirect methods (see discussion below on Use of the Analytical Balance).

Two final aspect of this experiment include the creation of a laboratory notebook (p 25, see below) and the submission of your report (see below). Be sure to save to disk a copy of your report and archive a copy on disk elsewhere. Update the archive file with each new laboratory throughout the semester.

Procedures

1. Prepare your laboratory notebook (see below).
2. Obtain a sample from your TA and record the code number into your laboratory book.
3. Place your sample into a weighing bottle (p. 36) and place the weighing bottle with mouth open into a clean 250 ml beaker. The beaker should be labeled with your name but you should never write or mark the weighing bottle with pen, pencil or other marker. Endeavor to make a quantitative transfer from your sample vial to the weighing bottle (lose no sample or leave no sample in the original sample vial).
4. Place the beaker with a watch glass cover (Figure 2-18) into an oven at 110°C.

5. After 30 minutes of heating, remove your sample from the oven and place the entire item (beaker and bottle with sample) into a desiccator (p. 36). Allow your sample to cool for 15 minutes.
6. Remove your cooled sample from the desiccator and using tongs, place the cap on the bottle. Using tongs, make a mass measurement of your sample. Record the mass of the weighing bottle with sample in your log book. Instructions on using the balance are given below.
7. Repeat steps 4 to 6 until the difference between successive measurements are ± 0.0003 g or better.
8. Place a clean medium porosity Gooch filter crucible (p35) in a beaker (as with step 3) and bring to constant weight. Record the mass.
9. Place your sample into a 100-250 ml beaker and record the mass of the empty weighing bottle. The weight of your sample is the difference between the empty weighing bottle and the weighing bottle with your sample (step 6,7)
10. Add 50 ml of distilled water to the beaker with sample and swirl the beaker to dissolve the powder (salt or sugar). You should see traces of metal in the beaker. Decant the liquid without losing any metal.
11. Repeat the washing and decanting of Step 10 twice more with fresh 50 ml amounts of distilled water in each step. Discard the supernatant in each step.
12. Quantitatively transfer the metal pieces from the beaker into the fritted glass Gooch crucible and wash the sample further with distilled water to insure all soluble solid is rinsed from the metal. Be sure to stabilize the suction flask with a ring stand.
13. Remove the Gooch crucible from the suction flask and place in a 250 ml beaker. Place the beaker and crucible into the oven at 110°C and bring to constant weight. The weight of metal is found by difference between the empty Gooch crucible (step 8) and the Gooch crucible with metal.
14. Calculate percent metal in your sample, complete your laboratory report and submit a printed copy of your report to the TA or professor by the due date. Be sure to propagate error in your calculation.

Sources of Error and Suggestions to Complete the Laboratory Successfully

1. Any glassware to be measured on a balance should not be touched with fingers or marked with pen, pencil, or other makers. Fingerprints and oils or dirt from fingers will introduce error to a measurement.
2. Samples or glassware not brought to constant weight will introduce potentially large error through buoyancy effects. Do not rush the drying step of your samples and glassware. Cool your sample in a dessicator.
3. Use the same balance throughout an experiment, tare your balance between each measurement and be sure to not lean on the bench during a measurement. Other balance errors may exist (pp 28-29).
4. You may have simple errors in calculations or you may include too many or too few digits (see section on significant figures pp 46-47).
5. Mistakes in keeping log book (p 25) including improper logging of the sample number or the sample code.
6. Impurities or dirt in the oven might fall into your sample if you do not have a watch glass on the beaker.
7. Be sure to use an oven at 110°C (not too hot or too cold). Be sure your weighing bottle is not capped during the drying step.
8. Watch that you do not loose metal bits during the decanting step.
9. You must have washed your metal bits free of any soluble solid.

YOUR LABORATORY BOOK

A laboratory book, when used well, will be substantially better than the use of scraps of paper to record your results. Such slips of paper may be lost or lead to transcription errors; loose papers including three ringed-binder or spiral bound notebook may also lead to confusion in experimental records and must not be used in Chem 371. Use an inexpensive but bound laboratory book available in the university bookstore or elsewhere.

At another level, your laboratory note book is a type of protection for you as a professional. Should you ever find yourself at the center of an accusation of falsifying data (see http://www.usdoj.gov/opa/pr/2002/September/02_enrd_536.htm and <http://www.bizjournals.com/tampabay/stories/2003/03/10/story8.html>), your laboratory book will be your only objective help in a lawsuit or other troubles. Thus, your notebook equals your reputation as a professional.

An example of a well-kept laboratory book is shown below. Note that these books need not always have perfect penmanship and that errors are never erased but corrected as shown below. Your laboratory book must have all pages numbered consecutively and a page should never be removed from the book. Failure to produce a notebook with proper accounting of an experiment will lead to an automatic scoring a laboratory to 0% since the notebook is the only objective evidence that your laboratory report is authentic. This policy will be in-force throughout the course and is not subject to appeal.

-5-

Title: Determination of Metal in Solid

Start Date: 030912 Friday

Reference: 371 Lab Manual page 10, 2003 version

Analyst: C.A. Eiceeman

Principle: $m_s + H_2O \rightarrow m_{ms} + \text{salt}$

$$\%m = \left(\frac{m}{m + m_{ms}} \right) \cdot 100$$

Sample Code: 15-AG

Results:

Constant

Mass of Glass-Filter	I-	28.8431 g			
	II	28.8265 g			
	III	28.8260 g			
	IV	28.8261 g	Final wt		
			a		

Mass of Sample : Sample + bottle - 40.7282 g

Bottle - sample - 31.3011 g

wt sample - 9.4281 g

Sample Extracted by water & metal isolated & washed

Constant

wt of metal in Filter	I.	35.2397 g			
	II	35.2364 g			
	III	35.2360 g			
	IV	35.2359 g	Final wt		

C.A. Eiceeman

YOUR LABORATORY REPORT

Unlike research reports, manuscripts, and other scientific writings, the reports which are due in this course should be simple and concise. The emphasis is in clear communication of quantitative analytical results without detailed discussions. The example below is a model for your laboratory reports throughout the course. Reports are due on the first day class meets after the laboratory has been completed. Reports may be refused and no credit given if the deadline is not met.

Example of a Laboratory report for Chem 371:

TITLE: QUANTITATIVE DETERMINATION OF METAL IN SOLID			
DATE STARTED: Aug. 26, 2003			
DATE FINISHED: Aug. 28, 2003			
ANALYST: G.A. Eiceman			
REFERENCE: pp. 38-42, Quant. Analytical Chemistry, D.C. Harris, 6 th Edition.			
UNKNOWN CODE: 54B21			
OBJECTIVE: Determine the amount of elemental metal in a solid mixture containing elemental or alloy metal and a soluble substance. The percent metal will be determined gravimetrically where the metal will be isolated from the sample matrix using solubility and washings of the sample.			
RESULTS:			
Constant Weight of Sample			
I	II	III	IV
2.5678 g	2.5624 g	2.5618 g	<u>2.5616 g</u>
Mass of Sample			
Mass of Weighing Bottle and Sample: 2.5616 g			
Mass of Weighing Bottle alone: 1.0253 g			
Mass of Sample: <u>1.5363 g</u>			
Mass of Metal			
Mass of Gooch crucible			
I	II	III	
6.8078 g	6.8064 g	<u>6.8062 g</u>	
Mass of Gooch crucible with metal			
I	II	III	
7.5274 g	7.5268 g	<u>7.5269 g</u>	
Mass of Metal: 7.5269 g – 6.8062 g = 0.7207 g			
Percent Mass of Metal in Solid:			
% Metal = (0.7207 g / 1.5363 g) x 100 = 46.91 ₁ %			
Error Analysis (±0.0003/±0.0003) results in ±0.0006 or ±0.06 ₂ %			
PERCENT METAL IN SOLID UNKNOWN 54B21 = 46.91 ₁ ±0.06 ₂ %			
Submitted:	Signatures		
<u>Sept. 4, 2003</u>	_____		
Date	J. Hancock		
Accepted:	_____		
_____	_____		
Date			

USE OF THE ANALYTICAL BALANCE

Adapted and modified from <http://chemed.chem.purdue.edu/genchem/lab/equipment/analytical/instructions.html#use>

General guidelines for use of an analytical balance

1. Always use the same balance in an experiment for best accuracy.
2. The balance should be clean
3. Never weigh a chemical directly on a balance (only chemicals in weighing bottles)
4. Do not lean or write on the bench surfaces near a balance.

We will be using Denver Instrument Company, Model 100A balances in Chem 371. Their website is: <http://www.denverinstrumentusa.com/>

Preparing the balance for use

Before using an analytical balance, insure that the balance is level. Look at the leveling bubble on back of the balance. If the bubble is not centered, turn the leveling screws on the bottom of the balance in front and back. Once the balance is leveled, close all the chamber doors and press the TARE bar on the front of the balance. After a few seconds, a row of zeros will appear. This indicates that the balance is zeroed and ready for use. The balance should be TARED before each use.

Approximate mass determinations

If you wish to weigh samples to only approximate values, you should not use an analytical balance as your first choice but rather a top loading balance with a range of ± 0.1 g. To make such approximate measurements you can place an appropriate weighing container on the balance and then tare the balance. The readout will read zeros with the container sitting on the pan. Add the substance to be weighed. Be careful not to spill chemicals on the balance. If necessary, you can remove the container from the weighing pan while you add the sample provided that no one presses the Tare bar before you weigh your sample. You will be able to obtain mass of your sample directly from the balance. This method should never be used for precise quantitative measurements (too many steps and too inefficient).

Precise quantitative weighs using an Analytical Balance

All mass determinations of high accuracy and precision should be made using mass by difference. You begin by adding more than enough sample for one or multiple measurements to a weighing bottle (not a beaker). After you bring your sample to constant weight, you are ready to weigh your sample(s). In a weight by difference method, you should have an idea approximately of the size of sample corresponding to the desired mass or weight. To make a weighing, you place the weighing bottle with sample on the balance, remove the bottle and spill out some sample into the intended vessel (for example, a beaker). Rapidly and directly return the bottle to the balance and note the new weight. The new weight, subtracted from the original weight is the weight of your sample. Note that you should not make multiple transfers from the weighing bottle for a single sample (too much error).

Cleaning and Leaving the Balance

When your measurement is completed, make sure you have properly cleaned any chemical spills on the balance. Doors should be closed. Do not move the balance as courtesy to others.

Version 1.2. August 2003, G.A. Eiceman, NMSU