

CHEMISTRY 3452 Quantitative Analysis Lab

***Note: Lab starts on FIRST WEEK**

Course Description: The CHEM 3452 lab course is to accompany the CHEM 3451 Quantitative Analysis. Various experiments are designed to utilize statistical treatment of data, sampling and transfer techniques, gravimetric and volumetric methods, titration analysis, electroanalytical and introductory instrumental analysis.

Course Objectives:

- To cultivate students' hand-on operation skills in field of quantitative analysis
- To introduce quantitative measurements in gravimetric, volumetric, electroanalytical and chromatographic separation.
- Understand factors that affect accuracy and precision of measurements and apply statistical analysis.

Lab Instructors

- 1) **Sairaman Seetharaman *** [SairamanSeetharaman@my.unt.edu]
(***Lab Coordinator**)
- 2) -----TBA-----
- 3) -----TBA-----
- 4) -----TBA-----

Lab Manual

Lab Manual will be provided. There will be a brief discussion of each lab at the beginning of the period. **The student will be expected to have read the lab manual before coming to class, since the discussion will focus on why, not how, the lab is done.**

Materials: Besides this lab manual, you will also need a lab notebook for class. You must have a writing utensil (pencil is not allowed) to record your data in ink. And eye protection, either safety glasses or goggles, is mandatory. Notebooks will be inspected at the end of lab session, and unannounced times during the semester, and graded according to completeness and organization.

Lab Reports: The last page of each lab handout is the lab report sheet, on which you will report your lab results. This information will come directly from your lab notebook (see below, lab TA will verify this at the end of each lab), and any blanks on the report must be filled in or explained. The completed lab report that gives concise summary of the results, correction and discussion are due at the beginning of the next lab period. Late

lab reports will be penalized by one point (20 points/lab), and no lab report will be accepted more than two weeks after the date due.

Lab Notebook: All students will use a lab notebook to record all data obtained in this lab. The notebook must be one in which the pages are permanently attached -- loose leaf notebooks are not acceptable. Recording data on scratch paper, paper towels, etc. before transfer to the notebook is expressly forbidden. Any student found using such scratch paper will have their lab grade for that lab lowered by 1 point (20 points/lab), and the scratch paper will be discarded.

Missing Lab: Arrive late over 15 minutes will be counted as absence. Missing lab will receive zero grade. No make-up lab unless permission obtained *in advance*. Medical absence requires proper doctor's statement.

Grading

The lab grade of CHEM 3452 will be calculated as follows

Laboratory report and notebook	75%
2 Quizzes	20%
TA Evaluation	5%

Notebooks will be inspected at the end of lab session, and unannounced times during the semester, and graded according to completeness and organization.

The "TA Evaluation" portion of your lab grade will reflect your attitude, preparedness, and safety-consciousness during lab.

Grading Scale

Final percent Average	Letter Grade
90 - 100 %	A
80 - 89 %	B
70 - 79 %	C
60 - 69 %	D
Below 60 %	F

The Chemistry Department believes in reasonably accommodating individuals with disabilities and complies with university policy established under Section 504 of the Rehabilitation Act of 1973 and the Americans with Disabilities Act (1990) to provide equal access and opportunity. Please communicate with your professor as to your specific needs and/or the office of Disability Accommodation (ODA) (Room 321, Union, 565-4323).

Academic Ethics: A high level of ethical conduct will be maintained in this course. Any evidence of an act of academic dishonesty during the exams will result in an automatic F

and expulsion from this course. Please adhere to University policies and the UNT Code of Conduct and Discipline with respect to academic ethics and honesty.

<http://vpaa.unt.edu/academic-integrity.htm>

CHEM 3452

LABORATORY SCHEDULE

Week of	<u>Lab #</u>	<u>Lab Title</u>
Start on 1st Week	1	Check-in / Safety-Use of Lab Equipment
(No Lab on Labor Day Week)		
3rd week	2	Gravimetric Determination of Calcium
4th week	3	Determination of Acid in Vinegar
5th week	4	Determination of Sodium Carbonate
6th week	5	Determination of Water Hardness
7th week		Quiz (Labs 1-5)
8th week	6	Potentiometric Determination of KHP
9th week	7	Determination of Fe by KMnO₄
10th week	8	Determination of Iodine using Iodate
11th week	9	Faraday's Law: Ni Electroplating
12th week	10	Gas Chromatography/Mass Spectrometry
(No Lab on Thanksgiving Week)		
14th week		Quiz (Labs 6-10) / Check-out

Lab Reports

The last page of each lab handout is the lab report sheet, on which you will report your lab results. This information will come directly from your lab notebook (see below), and any blanks on the report must be filled in or explained. The report sheets are due at the beginning of the lab period immediately following the completion of the lab. Late lab reports will be penalized by ten points, and no lab will be accepted more than two weeks after the date due.

Lab Notebook

All students will use a lab notebook to record all data obtained in this lab. The notebook must be one in which the pages are permanently attached -- loose leaf notebooks are not acceptable. Recording data on scratch paper, paper towels, etc. before transfer to the notebook is expressly forbidden. Any student found using such scratch paper will have their lab grade for that lab lowered by 10 points, and the scratch paper will be discarded.

Your lab notebook must always be up-to-date. Since you will not be recording data anywhere else, this should not be a problem. The TA will check notebooks during the lab period, and anyone found with an incomplete notebook for a previous lab will have their "Notebook" grade lowered by 5%.

The notebook will contain the following information in a clear, easy-to-read, understandable manner:

- A) A brief description of experimental procedure, or a flow chart.
This should be written in advance of the lab period, and is for your own use as an organizational aid as you perform the lab.
- B) All raw data, preferably recorded in data tables for easy reference.
- C) At least one example of every calculation.
- D) All conclusions (such as composition of unknown), and any reasons why lab results are not up to expectations (such as: "neighbor's experiment blew up all over my reaction vessel"). Results should be in tabular form, well labelled, and easy to understand by someone not familiar with your notebook.
- E) If your notebook is illegible, all conclusions will be assumed to be incorrect and graded accordingly.

Leave an empty page at the beginning of your notebook for a "Table of Contents". Fill it in as you complete each experiment.

Lab Clothing and Eye Protection

Eye protection is required by state law for everyone in a laboratory, regardless of whether they are actually doing anything or not. Goggles are strongly recommended since they provide more adequate splash protection. Any person who refuses to wear eye protection will leave the laboratory and take an automatic "0" for that lab exercise.

We will be using large quantities of acids and bases this semester. These chemicals tend to dissolve clothing (and flesh) with which they come into contact. It is advisable to consider any garment worn to lab as potentially disposable -- dress accordingly. Also: since most liquids tend to follow gravity after a spill, long pants and closed shoes are recommended.

Exercise caution when touching anything. It is especially unwise to sit or lean on the lab benches. If an acid or base has been spilled and left to evaporate, it will have left a residue that could easily irritate skin or clothing it contacts.

Graphing

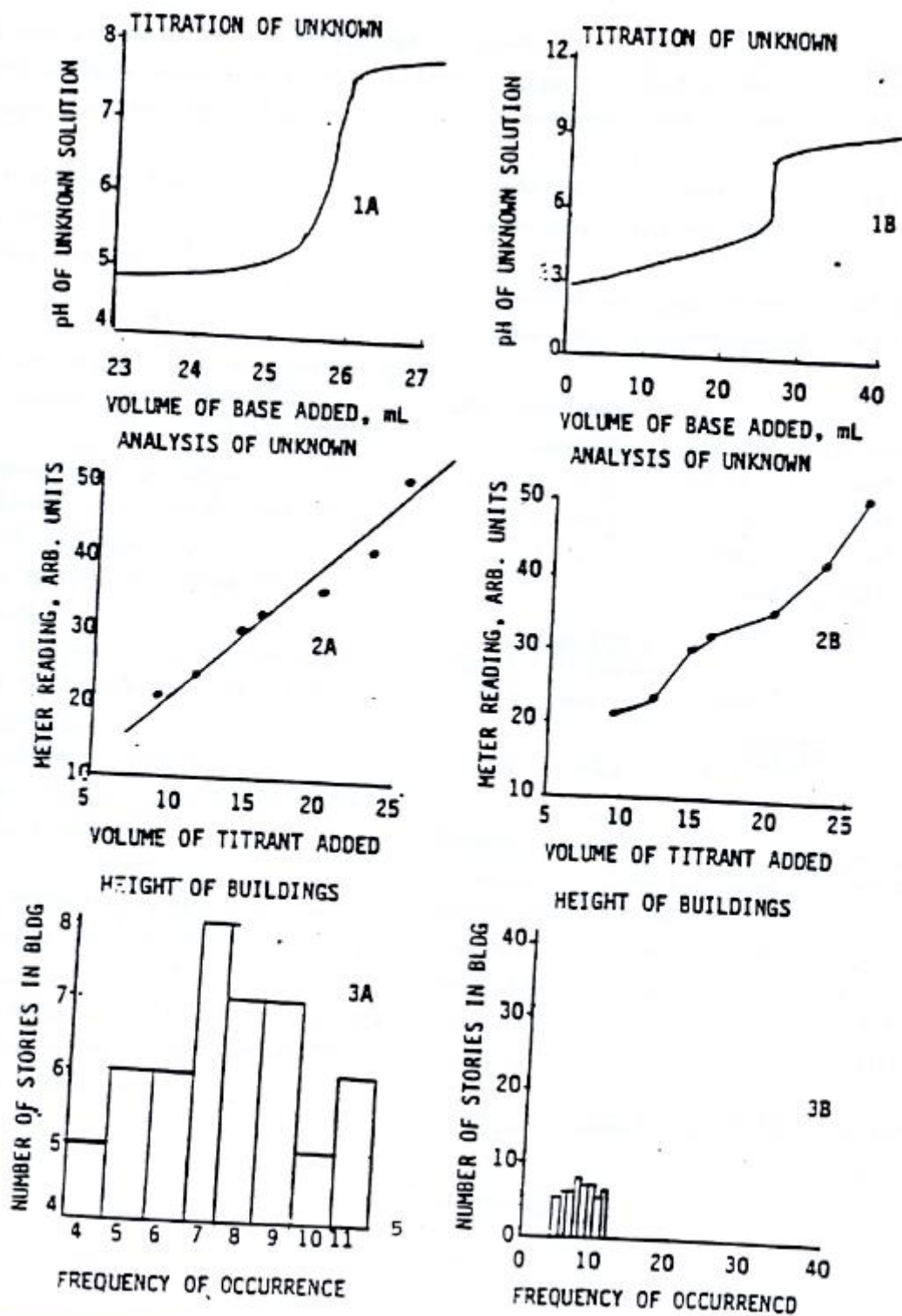
Several experiments in this lab will require the use of graphical methods of data analysis. When graphing continuous data (including most experimental results), a smooth curve should be drawn through the data points so that there are an equal number of points above and below the line. This is essentially a method of determining the average value of a function along the curve. A few other points to remember when graphing:

- A) Use as much of the graph paper as possible. Your graphs will be more readable and more accurate.
- B) If more than one curve is shown on the same sheet of paper, use different colored lines, different symbols for the data points (circles and stars, for example), or dotted versus continuous lines to differentiate the data sets. Make certain the difference is obvious, and provide a key to identify which is which.
- C) The x- and y-axes need not start at zero. Use only the parts of the axes which contain the domain and range of your data.

The following graphs illustrate these points, with the graphs on the left showing good techniques and those on the right showing poor techniques. 1A and 1B show the benefit of graphing only that portion of the graph which is of interest. Graphs 2A and 2B

illustrate the best way to draw a line through a series of data points (calculators can do this by a least squares program). Graphs 3A and 3B illustrate why you should use reasonable scales on both axes.

Place holder for Figure 1 to 3 A & B.



EXP. NUMBER 3.	EXPERIMENT/SUBJECT Determination of Acetic Acid in Vinegar	Place holder for lab report example (3 pages)		59
NAME	LAB PARTNER	LOCKER/DESK NO. Sep 17, 2007	COURSE & SECTION NO. Chem 2452-301	

1. Purpose: To study acid-base indicators in greater detail.
2. Objective: To select an appropriate indicator for the quantitative determination of vinegar in an unknown liquid solution.

3. Equipment:
~~2. Objective~~ 250 ml volumetric flask
 volumetric pipet
 250 ml Erlenmeyer flask

4. Procedure:

Use NaOH solution that has already been standardized against the primary standard KHP. ($\text{NaOH} = 40 \text{ g/mol}$)
 $\sim 0.2 \text{ g} \quad \sim 0.05 \text{ M} = \sim 10 \text{ g NaOH}$

- Obtain approximately 80 ml unknown.
- Use a volumetric pipet to transfer 25 ml vinegar sample to a 250-ml volumetric flask.
- Dilute to the mark with distilled H₂O. Mix thoroughly.
- Pipet 50 ml aliquots into 250-ml Erlenmeyer flasks, and add 50 ml distilled H₂O and 3-4 drops of phenolphthalein indicator.
- Titrate with the 0.05 M NaOH. (The endpoint signal is the first pink coloration that persists for 30 seconds.)
- Repeat the titration using a fresh 50 ml aliquot of diluted unknown solution, this time using 3-4 drops of methyl red indicator.
- Repeat the titration until all five indicators have used. (Note, it will be necessary to prepare a second 250 ml batch of the dilute unknown solution.)

5. Data:

Indicator	color Acid → color Base	Transition range (pH)
phenolphthalein	colorless → faint pink	8.0 - 9.6
methyl red	red → yellow	4.8 - 6.0
methyl orange	red → yellow	3.1 - 4.4
bromocresol green	yellow → blue	3.8 - 5.4
alizarin yellow	yellow → orange-red	10.1 - 12.0

NaOH Standardization:

#	Weight KHP	volume NaOH	M of NaOH
1	0.2071 g	0.0204 L	0.0497
2	0.2010 g	0.0191 L	0.0505
3	0.2159 g	0.0210 L	0.0503

$$\text{mean} = \frac{0.0500}{3}$$

$$\text{RSD} = \frac{0.0083}{0.0500}$$

$$M_{\text{NaOH}} = \frac{\left(\frac{\text{Weight of KHP}}{204.23 \text{ g/mol}} \right)}{V_{\text{NaOH}} \text{ (in liter)}}$$

$$\text{RSD} = \frac{S}{\bar{x}}$$

$$\text{FW of KHP} = 204.23 \text{ g/mol}$$

$$S = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$$

Determination of Unknown (Vinegar)

Indicator	volume (L)	M _{NaOH}	Acetic acid concn (wt. %)
phenolphthalein	0.086	0.0502	0.1012
methyl red	0.0716	0.0502	0.0935
methyl orange	0.026	0.0502	0.0313
bromocresol green	0.0498	0.0502	0.0576
alizarin yellow	0.094	0.0502	0.1133

$$\text{Weight \%} = \frac{M_{\text{NaOH}} \times V_{\text{NaOH}} \times 60 \text{ g/mol}}{25 \text{ ml}} \times \text{dilute factor}$$

$$\text{FW of CH}_3\text{COOH} = 60 \text{ g/mol}$$

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EXP. NUMBER 7.	EXPERIMENT/SUBJECT Determination of Acetic Acid in Vinegar	DATE Sep 17, 2007	60
NAME	LAB PARTNER	LOCKER/DESK NO.	COURSE & SECTION NO. Chem 3452 301

6. Formula & Calculations

Round 1 KHP = 0.2071 g

$$V_A = 0.2 \text{ mL NaOH}$$

$$V_B = 20.6 \text{ mL NaOH}$$

$$\Delta V = 20.4 \text{ mL} = 0.0204 \text{ L}$$

$$M_{\text{NaOH}} = \frac{(0.2071 \text{ g})}{(204.23 \text{ g/mol}) \cdot (0.0204 \text{ L})}$$

$$= 0.0497085 \text{ M}$$

$$\approx 0.0497 \text{ M}$$

Round 2

$$\text{KHP} = 0.2010 \text{ g}$$

$$V_A = 0.3 \text{ mL NaOH}$$

$$V_B = 19.8 \text{ mL NaOH}$$

$$\Delta V = 19.5 \text{ mL} = 0.0195 \text{ L}$$

$$M_{\text{NaOH}} = \frac{(0.2010 \text{ g})}{(204.23 \text{ g/mol}) \cdot (0.0195 \text{ L})}$$

$$= 0.05007 \text{ M}$$

$$\approx 0.0505 \text{ M}$$

Round 3

$$\text{KHP} = 0.2139 \text{ g}$$

$$V_A = 19.8 \text{ mL NaOH}$$

$$V_B = 21.0 \text{ mL NaOH}$$

$$\Delta V = 21.0 \text{ mL} = 0.0210 \text{ L}$$

$$M_{\text{NaOH}} = \frac{(0.2139 \text{ g})}{(204.23 \text{ g/mol}) \cdot (0.0210 \text{ L})}$$

$$= 0.05034 \text{ M}$$

$$\approx 0.0503 \text{ M}$$

$$M_{\text{NaOH}} = \frac{\sim 0.20 \text{ g KHP}}{204.23 \text{ g/mol} \cdot V_{\text{NaOH}} [\text{L}]}$$

$$\bar{M}_{\text{NaOH}} = \frac{0.0497 \text{ M} + 0.0503 \text{ M} + 0.0503 \text{ M}}{3}$$

$$= 0.0501667 \text{ M}$$

$$\approx 0.0502 \text{ M}$$

$$S = \frac{[(0.0497 - 0.0502)^2 + (0.0503 - 0.0502)^2 + (0.0503 - 0.0502)^2]}{3-1}$$

$$= 0.0004183013$$

$$\text{RSD} = \frac{S}{\bar{M}} = 0.008338504251$$

Phenolphthalein:

$$V_A = 0.3 \text{ mL NaOH}$$

$$V_B = 48.5 \text{ mL NaOH}$$

$$V_C = 0.3 \text{ mL NaOH}$$

$$V_D = 36.1 \text{ mL NaOH}$$

$$\Delta V_1 + \Delta V_2 = 48.2 + 36.8 = 85.0 \text{ mL} = 0.085 \text{ L}$$

$$\text{Weight \%} = \frac{(M_{\text{NaOH}})(V_{\text{NaOH}}) \times (60 \text{ g/mol})}{5 \text{ mL}} \times 10$$

$$= \frac{(0.0502 \text{ M})(0.085 \text{ L}) \times (60 \text{ g/mol})}{5 \text{ mL}} \times 10 = 0.1012 \%$$

metaph red:

$$V_A = 0.1 \text{ mL NaOH}$$

$$V_B = 49.0 \text{ mL NaOH}$$

$$V_C = 0.1 \text{ mL}$$

$$V_D = 29.0 \text{ mL NaOH}$$

$$\Delta V = 49.0 - 0.1 = 48.9 \text{ mL} = 0.0489 \text{ L}$$

metaph orange:

$$V_A = 0.0 \text{ mL}$$

$$V_B = 26 \text{ mL}$$

$$\Delta V = 26 \text{ mL} = 0.026 \text{ L}$$

Barium cresol green:

$$V_A = 0.2 \text{ mL}$$

$$V_B = 48 \text{ mL}$$

$$\Delta V = 48 - 0.2 = 47.8 \text{ mL} = 0.0478 \text{ L}$$

alizeron yellow:

$$V_A = 0.2 \text{ mL}$$

$$V_B = 50 \text{ mL}$$

$$V_C = 0.3 \text{ mL}$$

$$V_D = 40.5 \text{ mL}$$

$$\Delta V = (50 - 0.2) + (40.5 - 0.3) = 90 \text{ mL} = 0.09 \text{ L}$$

$$\text{Weight \%} = \frac{(0.0478 \text{ L})(0.0502 \text{ M})(60 \text{ g/mol})}{5 \text{ mL}} \times 10$$

$$= 0.0935 \%$$

$$\text{Weight \%} = \frac{(0.026 \text{ L})(0.0502 \text{ M})(60 \text{ g/mol})}{5 \text{ mL}} \times 10$$

$$= 0.0313 \%$$

$$\text{Weight \%} = \frac{(0.0478 \text{ L})(0.0502 \text{ M})(60 \text{ g/mol})}{5 \text{ mL}} \times 10$$

$$= 0.0576 \%$$

$$\text{Weight \%} = \frac{(0.09 \text{ L})(0.0502 \text{ M})(60 \text{ g/mol})}{5 \text{ mL}} \times 10$$

$$= 0.1133 \%$$

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7. Discussion:

The indicator I feel the most appropriate for the quantitative determination of acetic acid in vinegar is the bromocresol green indicator. Because vinegar consists primarily of 4% to 6% solution of acetic acid, and the acetic acid content ($\frac{1}{1000}$) of the bromocresol green is about 5.76%, which is in an acceptable range.

Selection of an inappropriate indicator would be a determinant (systematic) error because each indicator has its pH range of detecting acid ~~and~~ ^{content}. The far from the expected value the indicator you choose, the more systematic error you have, the higher the deviation from the true value.

good. to Aug 09/24/2007.

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