

ANALYTICAL CHEMISTRY 256 ROLE - PLAYING LAB

1998-1999 Academic Year

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St. Olaf College

Northfield, MN 55057

Introduction to Role-Playing and Laboratory Computing
Defining the Kinds of Role-Playing Responsibilities

Round-Robin Certification of Laboratory Glassware

Production Quality Control Lead Analysis
Statistical/Chemical Evaluation of Lead Data
Semi-Automated Weak Acid Titration
Graphical Analysis of Weak Acid Titration
Designing a Mock Robot Experiment
Executing the Mock Robot Experiment
The Incredible Edible Easter Egg Grass Advertising Dilemma
The Downsizing Dilemma
The Broken Pill Coating Machine Assembly Line Shutdown Dilemma
The Instrument Purchase Payment Release Dilemma
Closure

ROLES:

Manager: Selection of Certification Procedures - Verifying the Certifications
Chemist: Cleaning, Handling, and Using Volumetric Glassware
Software: Spreadsheet Setup and Inter-Company Data Telemetry
Hardware: Electronic Balance Computer Connection and Operation

OBJECTIVES:

Manager has to decide which pieces of glassware to certify by direct calibration, how to use the data to establish general error limits for later work, how to communicate the results to others in the lab, and how to divide the time between planning, data acquisition, and statistical analysis of the results. In addition, safety procedures have to be established and monitored for **Chemist** to use when cleaning the glassware. With results available from other companies, and some data from previous classes, good statistical results should be derivable that would let everyone know what acceptable tolerances are on the glassware and the way that it is used. This will require interaction between company **Managers** and electronic exchange of lab data and spreadsheets.

MANAGEMENT INTERVIEW:

The rationale selected for certification and calibration are at issue here, along with the way in which data between classes and companies were used to validate the approach. The way in which communication was maintained during these interactions is a crucial issue. The way in which a general tolerance statement is to be made for the whole semester needs to be established. Overall time management is at issue. Success in file transfer is up for discussion, along with how other company's data were used.

Manager should have available at the interview all spreadsheet pages showing the certification of his or her Company's glassware, plus have access to other Company's data that have been used in the round-robin certification process. Discussion about how such things as the t-test and the Q-test were or were not used are quite appropriate. Additional discussion about how to keep an electronic lab record is something that **Upper Management** will encourage **Staff** to carry out.

Introduction to Certification

The Electronic Laboratory Notebook

Until now, most of you have been encouraged, and occasionally even required, to keep a laboratory notebook, in which you enter both narrative and data, usually in ink. You likely have been told never to enter data on small scraps of paper (which, presumably, are easy to lose). This approach dates back to the 1940's, and has been dropped as a preferred method by some organizations due to the legal liabilities it poses.

For example, it is known from legal actions on silicone breast implants, tobacco addiction, agent orange issues, and asbestos suits that no lab records can be held as confidential or proprietary. All laboratory records can be subpoenaed by the courts at any time, regardless of the times or places at which they were initially recorded.

In simple terms this means that subjective, narrative comments and observations that traditionally have been part of the scientists' laboratory notebook (such as, for example, "This stuff looks really nasty.") can be taken into a court under entirely different circumstances than they first were recorded. It is likely that some of you, if not most, will find in the future that you will be discouraged from keeping narrative comments in a written lab notebook, and will instead be instructed, except in discovery oriented research situations, to only record data, simple explanations, and time and date at which the work was done. Other information, such as your professional opinions about the work or some of its results, likely will be communicated verbally to your immediate supervisor, who will determine in what form, if any, they are to be recorded.

This is not to say that there will be no more written laboratory notebooks. What it does mean is that there will be more electronic lab record keeping using tables and spreadsheets, coupled to verbal job performance evaluations, just as we do here with **Manager** and **Software**.

Thus, it will be up to **Manager** to keep an electronic lab notebook during the time the work is being done, to receive data continuously from **Chemist**, **Software**, and **Hardware**, and to ensure that everything is correctly entered into the electronic document. **Manager** will receive data from others in the Company as the work progresses (definitely not afterwards!). Thus, people in the Company will in fact take their data on small scraps of paper, deliberately, and as soon as it is available, give it to **Manager** to enter into the electronic record. The small scraps of paper will likely not get lost, because they will be 3M Post-It's[®], and will conveniently be stuck to the boards available to all over the benches.

Data still will have to be written down. Data still can be lost (by electronic means). And loss of data, electronically or otherwise still will mean a failing grade for the experiment. But, in our lab, it will be up to **Manager** to keep the electronic lab records, and all communications with **Upper Management** will be in the Management Interview.

There are several ways to keep electronic records. Microsoft Word has as one of its strengths an excellent system of tables. A table can easily be made and edited in Word, and can be converted back and forth from text. Data can be dragged and dropped into a table. Another very attractive possibility is the Excel spreadsheet. Art work and sketches can be brought into the Excel sheet, and data can be entered, operated on, plotted, charted, and taken into other documents quite well. **Manager** can determine which method to use. The Company computers have all been licensed to carry Microsoft Office 98, so both Excel and Word will be available.

While Excel is easy to use, it does require some advanced design. **Manager** and his or her staff should be sure to decide before the lab begins what design to use. For help here, each of the experiments in this lab manual will have example sheets in them. Good work from previous classes also will be available on the server. It will be up to **Manager** to look at these data and decide if and how to use them. **Upper Management** also expects collaboration between Companies as the lab progresses, and there is no reason not to share spreadsheet designs as long as appropriate and explicit credit is given to the originators thereof.

Spreadsheet Calculations

Certification of the laboratory glassware you will be using is the start of that long process of **error detection** consequentiality that will enable **Manager** to tell if certain steps taken in an analysis **could have been responsible** for a suspected result. In all probability, no one step will be completely responsible for a particular result, good or bad. More like is the combination (or **accumulation**) of steps, with some being more **consequential** than others. Thus, where you may have learned elsewhere about "error analysis", I suggest that detection of step consequentiality by direct measurement and computer based accumulation is the more useful approach for **Manager** and **Software** to interact with in this laboratory.

An example Excel spreadsheet follows to illustrate the electronic lab record:

Example of a Spreadsheet Used to Hold the Data and Do Calculations					
Prepared by Software, Used by Chemist, Approved by Manager					
	Burette	Flask	pipette (10 mL)	pipette (25mL)	
glass type	EXAX	EXAX	EXAX	EXAX	
capacity (mL)	50.0	100.0	10.0	25.0	
Baro. P.	738.0	738.0	738.0	738.0	
Temperature	29.3	25.3	29.1	29.2	
wt. catch vessel	22.4475	64.8465	21.4520	22.2360	
Delivery Wt. A					
1	61.8349	164.3311	31.3797	47.2098	
2	61.8353	164.3319	31.3798	47.2102	
3	61.8353	164.3310	31.3800	47.2099	
Delivery Wt. B					
1	56.7911		41.3027	72.0848	
2	56.7912		41.3023	72.0849	
3	56.7907		41.3020	72.0845	
Net Del Weight A					
1	39.3874	99.4846	9.9277	24.9738	
2	39.3878	99.4854	9.9278	24.9742	
3	39.3878	99.4845	9.9280	24.9739	
Average net wt. A	39.3877				
Net Delivery Wt. B					
1	34.3436		9.9230	24.8750	
2	34.3437		9.9225	24.8747	
3	34.3432		9.9220	24.8746	
Average net wt. B	34.3435				
Ave. Net Del wt.		99.4848	9.9252	24.9244	
std. Dev.				0.0029	0.0540
%Rel St. Dev.			0.0003	0.0022	

(Continued on the next page)

	Burette	Flask	pipette (10 mL)	pipette (25mL)
glass type	EXAX	EXAX	EXAX	EXAX
capacity (mL)	50.0	100.0	10.0	25.0
Baro. P.	738.0	738.0	738.0	738.0
Temperature	29.3	25.3	29.1	29.2
Correction factor	0.4924	0.4008	0.4860	0.4898
Correction scale				
A	0.4000	1.0000	0.1000	0.2500
B	0.3500			
Net add Correction				
A	0.1970	0.4008	0.0486	0.1225
B	0.1723			
Corrected Volume				
A	39.5846	99.8856	9.9738	25.0469
B	34.5158			
% Relative Error		0.11 ⁴⁴	0.002 ⁶	0.001 ⁹

Lab Measurements

A set of **Standard Operating Procedures** are included as part of this experiment for **Hardware** and **Chemist** to use when actually doing the hands-on manipulations needed to make the certification measurements. Thus, it is not necessary to introduce what they will be like in detail here. But, there is one very unique measurement step that does require introduction. That is the use of **weight** as the primary liquid measuring quantity.

Weight is related to volume by density. **Chemist** would be well advised to review basic definitions of density from his or her first year chemistry or the analytical chemistry texts early on in this experiment. It is a basic concept, but often is confusing when used for certifications.

Clearly, if liquids are to be measured by weight, as opposed to volume, then it is density and temperature that become the two new variables in the measurement process.

We will be working with two kinds of volumetric glassware. One kind is designed "**to contain**" a certain amount of liquid. An example is the **volumetric flask**, of which **Manager** may decide to certify the 100 mL variety. The second kind is designed "**to deliver**", examples of which are the **volumetric pipette** and the **burette**. Detailed instructions are available for certifying the 25 mL pipette and the 50 mL burette, but **Manager** again may decide to adapt these instructions to other kinds of glassware in the Company drawers.

Weight will be measured using one of two electronic balances, both of which can (and probably always should be) connected by their **serial** data ports to a laboratory microcomputer. **Manager** has to interact with both **Hardware** and **Software** on this, since the connection will require the use of a LabVIEW program that **Software** will need to operate to "log" (jargon for acquire slowly) data into a text file that later can be **imported** into **Excel**.

BE CLEAN!

A key issue in the lab measurements is the deceptive one of "glassware cleanliness". How can anyone get excited about clean glassware? The answer lies in realizing that all volumetric glassware (as opposed to beakers and dishes) is calibrated either **to contain** or **to deliver** with an unbroken film of water flowing uniformly down its sides. Indeed, this is an actual **design** step!

So, when the film of water that drains, say, down the barrel of a pipette "breaks" into tiny droplets, due to changes in surface tension at the liquid-glass interface, then the amount of liquid delivered actually changes. This is what we call "dirty" glassware, and the amount of error introduced in a method depends totally on how dirty it is!

An issue related to the above is "drying" glassware. If, say, a volumetric flask is dried by blowing air into it from the air jets in the lab, it is almost a certainty that some oil and grease from the line will be blown into the flask, and form a spotty film on the inner walls. While the walls may appear clear, this does mean they are clean. Unless they are grease free, the flask will "spot" when draining a water film, and its calibration will be lost! Clear and clean mean two different things.

The only way to dry glassware is with a gentle stream of warm, dry air, and such hot air drying devices are available for each Company and over the sink on the South wall. But, it takes more time to dry glassware properly, so it soon becomes a management call.

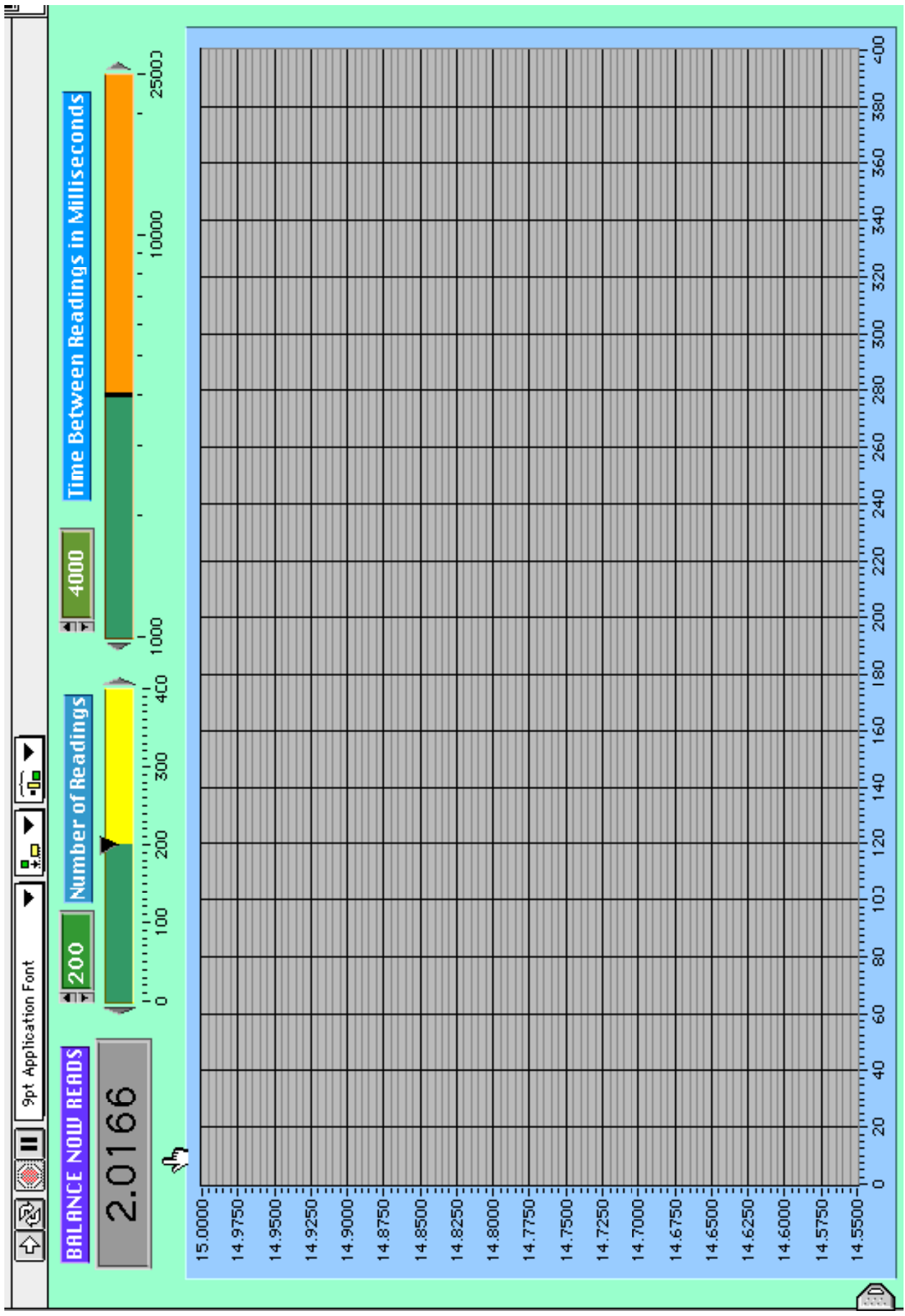
Chemist will clean the glassware. To do this, **Chemist** must follow the instructions listed in this write-up, and be especially conscious of the proper safety techniques. In addition, the cleaning solution of "alcoholic KOH" available to **Chemist** in the hoods will etch the volumetric glassware if it is left in contact with the glassware for more than 5 minutes in its concentrated form. This will require **Manager** and **Chemist** to coordinate the timing when the glassware is cleaned, so that it can be rinsed immediately and used before it gets dirty again. Either **Staff** or **Upper Management** will give a tutorial on using alcoholic KOH if requested.

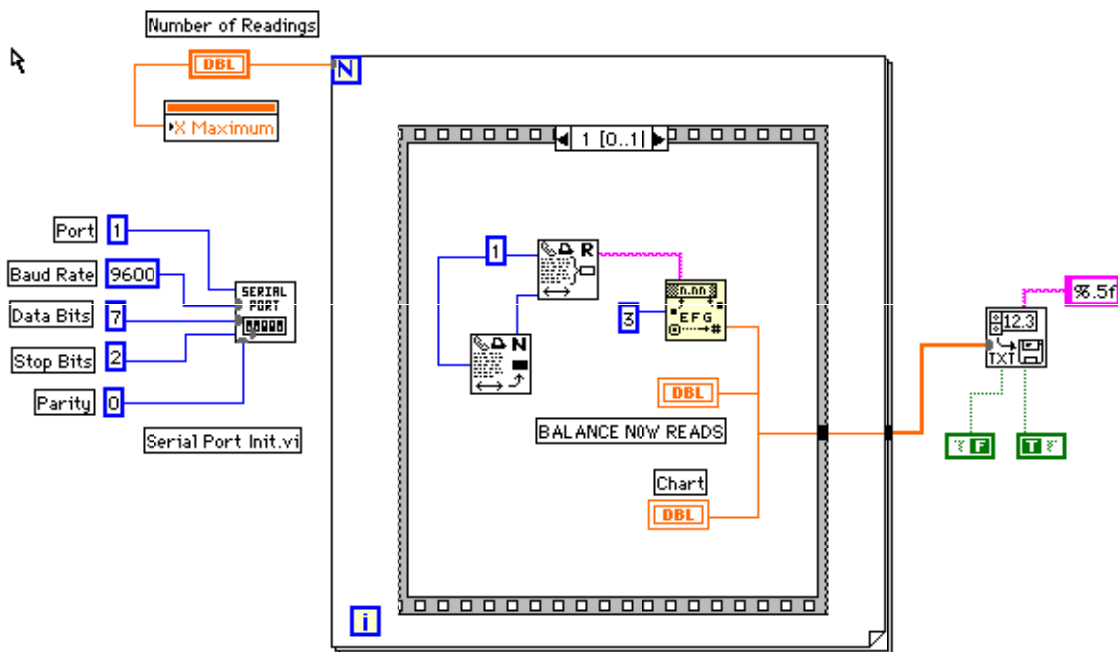
Hardware has the task of running the balances in this session. It seems trivial, and occasionally is pretty routine, but the results can be disastrous for the rest of the semester if done improperly.

Hardware also has the task of soldering the RS-232 "serial" cable together to connect the analytical balance to the serial port of the Company Mac microcomputer. Pin connections for this have been given in the previous experiment write-up (Introduction to Company Responsibilities). Parts will be furnished in the lab, and a short tutorial presented by **Staff** or **Upper Management** at the start of the period.

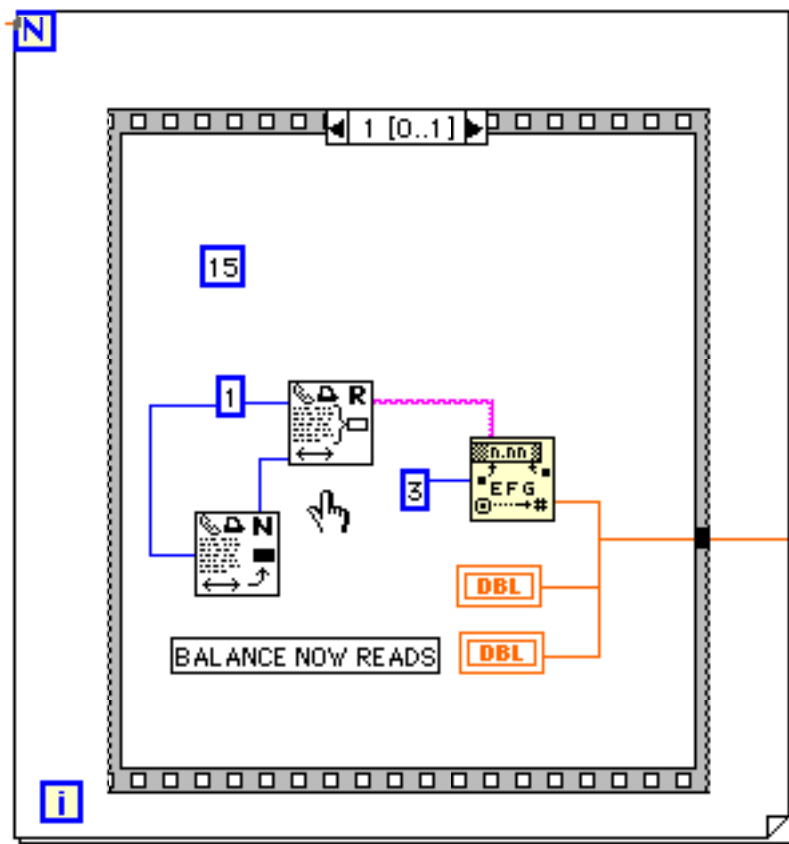
There is also the need for **Hardware** to collaborate with **Software** to decide how to handle the data. The simplest approach is for **Software** to just manually key into an opened spreadsheet the readings that **Hardware** comes up with from the display on the front of whichever electronic balance is used. An alternate approach is more automated. If the automated approach is taken (**Manager** decides) then **Software** will have to run the LabVIEW VI shown on the next page to log the data session to a file that will be stored on the hard disk as a named file which later can be opened as an Excel spreadsheet.

These LabVIEW programs are not difficult to write, or to operate. Most of those used in Chemistry 256 were written in Chemistry 378. If **Software** wants to try designing a new front panel, altering the color scheme, or adding more features to the existing VI's, **Manager** should arrange for some tutorial sessions with **Upper Management** to that end.





Back panel diagram for the balance reader showing just the read function.



Interpretations

What finally has to come out of this experiment, from **Manager** to **Upper Management**, is an estimate of how much error can be expected from using clean and dirty volumetric glassware, whatever experiment and whatever piece of volumetric glassware is used. **Manager** should look ahead in the lab schedule to locate who will be managing the "Structural Unemployment" management dilemma, since that dilemma specifically uses the data from this experiment in helping **Manager** reach a personnel decision.

First, **Manager** is going to have to go through the lab glassware complement (the lab drawer) and decide which pieces to certify, so that the data that come back are actually useful for predicting what errors can be expected from the other pieces. Let's explore some possibilities.

Management Decisions

Manager may realize that titrations are ahead, just by looking at the upcoming lab schedule. Reviewing the contents of the drawer, s/he would see a burette listed. Looking at the appended detailed discussions, s/he would see detailed directions for **Hardware** to use in calibrating the 50 mL burette. This would seem like a natural choice then as one piece to calibrate.

Similarly, looking at the course text for gravimetric methods in general, **Manager** would find pipettes used everywhere. But, here, there are a wide variety of sizes available from which to choose. Should the detailed directions be followed, and only certify a 25.0x mL pipette? Could the resulting error be extrapolated to, for example, a 10.0x mL device, or a 100.0x mL device? Should two pipettes be certified to test this? Should one Company do one, and another? Do you, **Manager**, trust another Company's results?

What about volumetric flasks? Is there really a choice here, considering the weight capacity of our analytical and top loading balances? Could you, for example, certify both the 100.x mL flask and the 250.x mL flask, and extrapolate the **relative error to other flasks**?

The data in previous lab Company files should help **Manager** make design decisions. They also can help **Software** in designing spreadsheets, and **Chemist** deciding how to approach the work. An example of one of these, submitted to either **Staff** or **Upper Management** by electronic mail, follows for written reference.

From hazen Mon Feb 25 17:33:15 1991
 Received: by .laichi.uucp;
 id AA02362; 5.59/LAI-XENIX-1.0; Mon, 25 Feb 91 17:33:11 CST
 From: Brett Hazen - Chem 55/56 <hazen>
 To: hazen, walters
 Subject: Results of LauraT's Feb21
 Date: Mon Feb 25 17:33:07 1991

CERTIFICATION OF LAURAT's LABORATORY GLASSWARE

Report Submitted at 5:25 PM on February 25, 1991

Manager: Brett Hazen

Chemist: Paul Jackson

Software: Claire Jordan

Hardware: David Kell

Experiment run on: February 21, 1991

After a brief faculty meeting we decided to pool our glassware, select the best vessels and then keep them in one drawer which will be used for all of our experiments. We exchanged combinations, in case we need to access our glassware and the one individual has not reached the laboratory yet. **Chemist** checked glass for cleanliness and cleaned them. **Software** learned how to transfer files between environments and how to use the spreadsheet Excel-**Hardware** and **Software** worked together to get our electronic scale and PC linked to electronically collect data. **Hardware** gave a brief demonstration on how to use the scales and how to use two-point calibration on the pH meter. **Chemist** gave a brief demonstration on the use of KOH alcohol as a glass cleaning agent. The company participated in formatting the Excel spreadsheet. Time was also spent familiarizing team members with computing system.

Overall our results were very good with %RSD's of 0.0197% to 0.0935%.

CERTIFICATION OF ANALYTICAL GLASSWARE LAURAT COMPANY

Data Collected on Feb 21, 1991 at 4:30 pm CST on LAURA

Type of Glassware KIMAX

CERTIFICATION OF PIPETTES

Part A:

Nominal Capacity of Pipette	10 mL (Graduated)
Barometric Pressure when Weighing	732.6 mm Hg
Room Temp	23 C

Water Temp	25.5 C
Tare Weight of Catch Vessel	28.3077 g

Test Delivery Weights		
	1	38.2237 g
	2	48.1492 g
	3	58.0520 g

Net Delivery		
	1	9.9160 g
	2	9.9255 g
	3	9.9028 g
Correction Factor		0.4047
Scale Factor		0.1000
Net Correction		0.0405
Corrected Weights		
	1	9.9565 g
	2	9.9660 g
	3	9.9433 g
Average Weight		9.9552 g
Standard Deviation		0.0093
Relative Standard Deviation		0.0935 %
Volume of Vessel at T and P		9.9552 mL

Part B:

Nominal Capacity of Pipette	50 mL
Barometric Pressure when Weighing	732.6 mm Hg
Room Temp	23 C

Water Temp	25.5 C
------------	--------

Tare Weights of Catch Vessels		
	1	28.5375 g
	2	27.7685 g
	3	27.7658 g

Test Delivery Weights		
	1	78.2822 g
	2	77.5384 g
	3	77.5555 g

Net Delivery		
	1	49.7447 g
	2	49.7699 g
	3	49.7897 g

Correction Factor		0.4047
Scale Factor		0.5000
Net Correction		0.2024

Corrected Weights		
	1	49.9471 g
	2	49.9722 g
	3	49.9921 g

Average Weight		49.9704 g
Standard Deviation		0.0184
Relative Standard Deviation		0.0369 %

Volume of Vessel at T and P 49.9704 mL

CERTIFICATION OF VOLUMETRIC FLASK

	LAURA	DEANO
Nominal Capacity of Vessel	100 mL	100 mL
Barometric Pressure when Weighing	732.6 mm Hg	732.7 mm Hg
Room Temp	23 C	24.5 C
Water Temp	25.5 C	24.5 C
Tare Weight of Empty Vessel	66.2483 g	68.95 g
Net Delivery Weights		
	1 165.7939 g	168.36 g
	2 165.7690 g	
	3 165.8171 g	
Net Delivery		
	1 99.5456 g	99.41
	2 99.5207 g	
	3 99.5688 g	
Correction Factor	0.4047	0.4047
Scale Factor	1.0000	1.0000
Net Correction	0.4047	0.4047
Corrected Weights		
	1 99.9503 g	99.91
	2 99.9254 g	
	3 99.9735 g	
Average Weight	99.9497 g	
Standard Deviation	0.0196	
Relative Standard Deviation	0.0197 %	
Volume of Vessel at T and P	99.9497 mL	99.91 mL

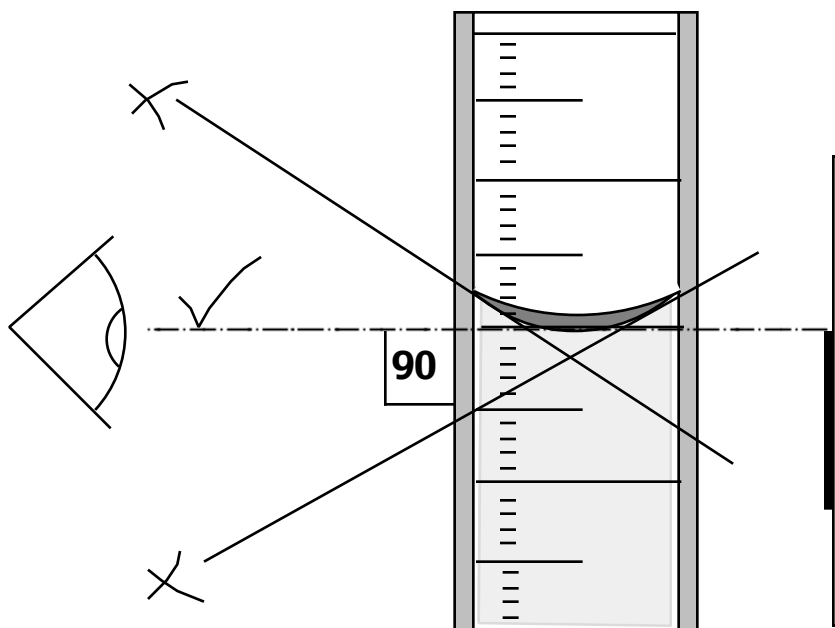
Respectfully Submitted --- Brett Hazen

Detailed Directions

Preliminary Operations

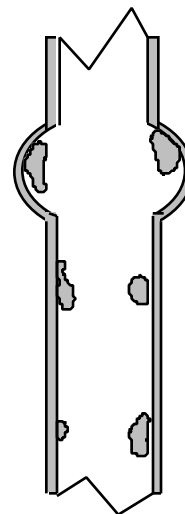
Preparing the Glassware for Certification

1. Locate the 50 mL burette, 25 mL pipette, and 100 mL volumetric flask in your lab drawer (if that is what **Manager** has decided to certify). Inspect them for damage, paying particular attention to the condition of the tip of the burette and pipette. The ground glass end should be intact.



2. Verify to your own satisfaction that you recall how to use a burette reading card to read the meniscus of the burette at various settings. Check yourself against the other persons in the lab. If you have any doubts, seek individual instructions now. Observe the diagram at the left for the proper and improper ways to sight the meniscus using a blackened stripe burette reading card for review.

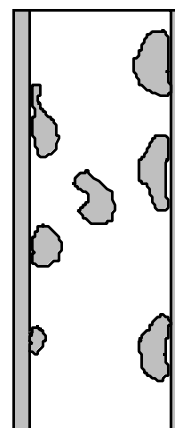
3. Inspect the 100 mL volumetric flask to ensure that it is clean. Fill it with deionized water and note that the neck near the etched mark, as well as the rest of the flask, drains cleanly without spotting. If not, clean it now with soap solution. Rinse it thoroughly with deionized water and repeat the process until it does drain without water spotting. If the flask will not clean with soap and deionized water, **Chemist** may use alcoholic KOH solution. Detailed instructions for using this solution are at the end of the experiment.



4. Repeat the cleaning for the pipette and burette.
5. Dry the 100 mL volumetric flask by using the hot air device near your Company work area or next to the sink. **Do not use acetone**, or air from the jets in the hoods. Acetone will contaminate the environment, and the air in the hoods and on the benches has grease suspended in it.
6. Place the flask aside. Stopper it with the appropriate ground glass stopper (or equivalent) for tare weighing on the **top loading** electronic balance. Do not get the inside of the flask wet before tare weighing.
7. The burette must be filled, checked for air bubbles, cleanliness, and stopcock leaks before it can be certified. The next steps refer to this process.

8. Set up the burette with appropriate clamps at a convenient eye level. Fill the burette at least 0.5 mL above the zero mark with deionized water.
9. Wipe the outside of the burette clean of any spills. Use a piece of filter paper to blot the tip.
10. Adjust the clamp used to make sure the meniscus is level and can be read easily with a blackened burette reading card.
11. Flush any and all air bubbles from the burette tip, stopcock bore and the burette barrel by delivering several brief "spurts" into a waste vessel. If any bubbles persist, repeat the process accompanied by sharp tapping of the tapered end of the burette with your finger.
12. If necessary, refill the burette. Slowly drop the liquid level to about 0.5 mL below the zero mark. Close the stopcock and accurately read the meniscus. Record the reading.
13. For a period of approximately 15 minutes, periodically check the level of the meniscus in the burette. If the stopcock is properly seated, the level will not have changed by more than your reading error (e.g., 0.03 mL) If it has, disassemble and reseal the stopcock. Check with **Manager, Staff, or Upper Management** for interpretive advice.
14. To test for cleanliness of the burette, observe that it drains a uniform film of water with no streaking or formation of spots on the walls under the following two tests:

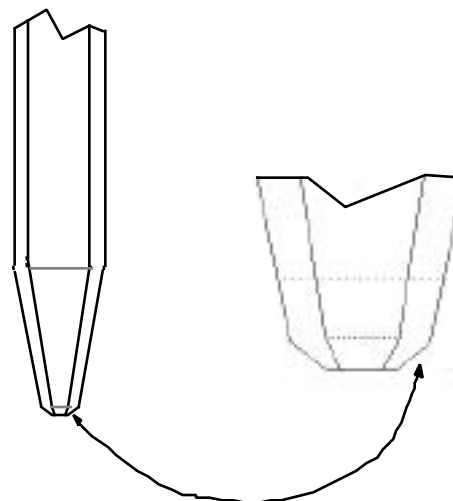
- Fully open the stopcock and rapidly drain about 40 mL into a waste vessel. Close the stopcock and closely inspect the inner walls of the burette for any breaking in the residual water film (including at the top for at least an inch above the zero mark).
- Refill the burette and repeat the above for a slow draining rate, e.g., 10 mL/min. Observe the film just above the meniscus as it slowly falls. No breaking or spotting should occur.



15. If the burette drains cleanly, it may be set aside for certification. Fill it with deionized water and put a cork in the top. If it does not drain cleanly, then it will be necessary to scrub it with a burette brush and soap solution. Be gentle. Seek advice if you are unclear on this. Following cleaning, rinse the burette several times to remove the soap solution, and then to re-check it for drainage as described above.

16. It also is important to check the pipette for cleanliness and uniform drainage in a manner similar to the burette. The next steps refer to that process.

17. Closely inspect the tip of the 25 mL pipette. There should be no chips in the ground glass surface that extend over and into the circular bore through which liquid exits. If there is any question about this, ask. Observe the diagram at the right for guidance.

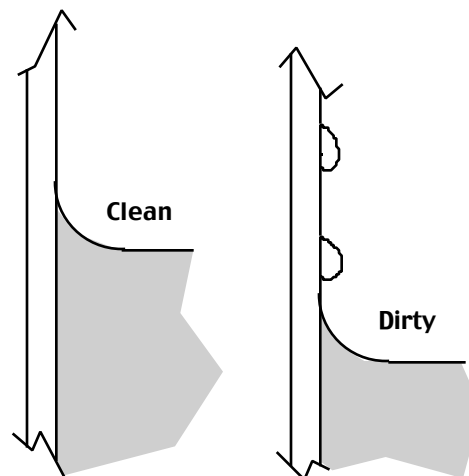


18. Fill a 400 mL beaker approximately 3/4 full of deionized water to use a reservoir to pipette from. Set aside a 250 mL beaker to pipette into.

19. Using a pipette bulb, fill the pipette with deionized water from the 400 mL beaker to at least two inches above the mark. If for any reason you are still uncomfortable with the motor skill aspect of this, now is the time to ask **Manager, Staff, or**

Upper Management for practical tips.

20. As a self-teaching demonstration, hold the pipette over the waste beaker and jar it slightly with the palm of your free hand (be gentle). Notice that a small "spurt" occurs and that an air bubble appears in the tip. This is what you do **not** want to happen when you are using the pipette for a quantitative transfer; thus, you will have to move it without jarring. Practice moving the pipette around the lab bench area, held vertically and full, until you can do it smoothly enough to avoid this kind of spurting.
21. Refill the pipette (if necessary) to at least two inches above the mark. Allow it to drain freely into the waste beaker. While it is draining, watch the meniscus closely.
22. If the pipette is clean, then the water film that forms behind the meniscus as it drains will be uniformly smooth, without visible streaking or breaking up into spots. If the pipette is not clean, this will not occur, and it will be necessary to use deionized water and a soap solution to clean it. (See step 26.). Use the figure below for guidance.
23. The pipette should be filled and drained several times, and at different rates. At least once it should be held still above the waste beaker for a minute or so to be sure that the water film will not break.



Procedure for Cleaning Glassware with "Alcoholic KOH"

"Alcoholic KOH" is a cleaning solution that is made by dissolving 30 grams of solid KOH in a liter of ethanol. Its action as a cleaning solution comes from the KOH lightly etching the glass, releasing any grease on it to the ethanol environment where it usually is soluble.

The solution is hazardous, and must be handled only in the hood. **Chemist** is the person who must handle the solution. To do this, the proper safety protection must be worn. Clothing should be protected with a lab coat. "The St. Olaf College Chemistry Department Stockroom Policy" on lab coats is that they are only available to course instructors (at the time of this writing). If this is still in effect when **Chemist** has to clean glassware, then **Manager** will have to negotiate a temporary loan before the lab work begins (also, see **Upper Management** for the latest developments in this policy).

Eye protection is mandatory here, just as it is elsewhere in the lab. It is especially important though that the glass shield that forms the hood door be partially lowered, in part to prevent any upward splashes from leaving the hood.

Chemist should protect his/her hands by wearing a fresh pair of disposable polyethylene gloves. A box of these "DisPo" gloves should be available at each hood where the cleaning will be done.

Several small beakers, generally in the 150 mL size, should be in the hood area. A towel or two also will be needed. At least two of the blue pipette bulbs must be present. There should also be a 600 mL beaker approximately 3/4 filled with deionized water, and another of approximately the same size that is empty (to catch waste). A plastic wash bottle filled with deionized water also is a **necessity**.

Cleaning a Pipette

Consider the procedure for cleaning a pipette. Transfer about 75-100 mL of the alcoholic KOH to a 150 mL beaker. Immerse the tip of the pipette well below the top of the liquid level of the alcoholic KOH solution, and, using a blue pipette bulb, draw it very slowly up into the pipette. Allow it to pass the mark, by at least a centimeter or two. Release the vacuum from the pipette bulb, and slowly allow the viscous alcoholic KOH solution to drain back into the beaker. This delicate job has to be repeated at least three times to be effective on a dirty pipette; once may work if the pipette has been recently cleaned and just got dirty through use with deionized water.

When the above has been completed, **thoroughly** rinse the pipette. This is done by first drawing up water from the 600 mL beaker and flushing out the pipette several times. This, however, only dilutes the alcoholic KOH residue. After this is done, the pipette should be moved over the large, empty waste beaker and flushed from the top with the wash bottle. This is done by putting the narrow tip of the wash bottle into the top of the pipette, and gently flowing deionized water into the pipette. Rotating the pipette at the same time will ensure that all sides of the "bulbed" part are flushed.

Cleaning a Volumetric Flask

Consider next cleaning a 100 mL volumetric flask. The procedure here must of necessity be different than that for the pipette since liquid cannot be pulled into a volumetric flask by suction.

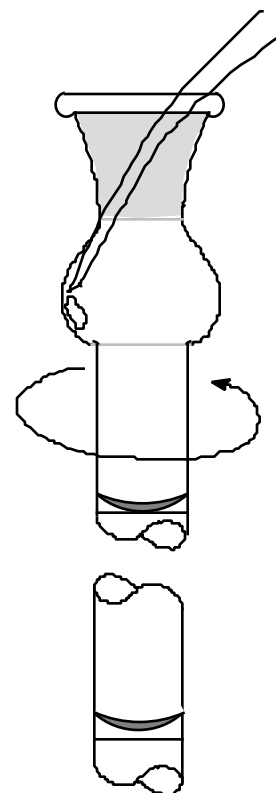
Start by transferring about 50 mL of alcoholic KOH into a 100 mL beaker. Then with slow deliberate care pour the alcoholic KOH into the volumetric flask, slowing rotating the flask at the same time so that the entire inner wall area of the neck is wetted by the solution. When all of the alcoholic KOH has been poured into the flask, slowly transfer it back to the original beaker, again slowly rotating the flask as the solution is poured out of it. The idea again is to thoroughly wet the inside of the flask.

When the flask has been filled and drained 2 or 3 times, use the wash bottle to wash down the inner neck with deionized water, and discard the water waste into the large waste beaker. Return the initial (now used) portion of the alcoholic KOH to the original container (it can be used many times so there is no need to discard it). The volumetric is now ready to be carried to the sink and rinsed several times under the deionized water tap.

There are other variations on these two procedures. They are best described by a demonstration, which **Upper Management** will give **Chemist** as the need arises.

Certification of the 100 Milliliter Volumetric Flask

1. Locate the dry, stoppered 100 mL volumetric flask. Wipe the outside of the flask clean of grease and other debris with a towel. The flask should be clean and dry, inside and out, before it is initially weighed.
2. Prepare a clean and dry place on the lab bench at which to work. Once the flask has been weighed empty, it must not be set in a place where it will pick up solid or semi-solid debris.
3. When carrying the flask, it is a good idea to handle it mainly by the top of the neck. This is not to prevent fingerprinting, but to avoid changing its temperature.
4. When **Hardware** has indicated that a balance is available, do the following before weighing the empty flask:
 - Clean the balance pan with a tissue so that it too is dry.
 - Weigh a dime or a quarter several times to get an idea of the precision of the balance and to become familiar with how to use the simple data logging procedure **Software** has implemented.
5. Weigh the empty flask at least three times, each time picking it up and moving it to a different place on the balance pan. Use the average of the three as its tare. Use whatever program **Software** has prepared to acquire the data and calculate the average.
6. Fill the flask about a quarter full with deionized water. Slosh the water about to wet all portions of the inside of the flask, including the top of the neck above the mark. Drain the flask over a sink, wipe the outside dry and make sure it is still clean enough to drain a film of water without breaking. If not, it will have to be cleaned, dried, and re-tared before continuing.
7. The flask may be filled with a plastic wash bottle in the manner shown in the diagram below. Make sure to rotate the flask so the upper neck is uniformly wetted. Fill the flask to slightly above the mark.
8. The level of the meniscus will rise above the mark as the neck drains. Insert a thermometer into the flask and wait a minute or so for the draining to finish and thermal equilibrium to occur. Read and record the temperature.
9. Remove the thermometer. It will take some water out by adhesion. Thus, readjust the liquid level to bring the bottom of the meniscus into coincidence with the mark. Remove or add water as needed with a long tipped dropper. If more than a mL is added, again measure the temperature.
10. Stopper the flask and weigh it using **Software's** program on one of the electronic top loading balances. Obtain at least three weightings to allow some averaging to compensate for handling and position on the balance pan.
11. Unstopper the flask and remeasure the temperature of the water well into the center portion of the flask. If this reading differs from the former by more than 0.5 degrees, use the average of the two, otherwise use the latter.



12. Following the calculation procedure shown below for a pipette, calculate the volume contained in the flask. The procedure used here is fine for certifying a flask, but should not be used when preparing a standard or the like. In that case, water should never be removed from the flask.

Example Calculation Procedure for Volumetric Glassware

Type of Glass	EXAX
Nominal Capacity of Pipette	25 mL
Barometric Pressure when Weighing	740 mm
Temperature when Weighing	21.5 °C
Tare Weight of Catch Vessel	13.6920
Test Delivery Weight #1	38.5900
Test Delivery Weight #2	38.5798
Test Delivery Weight #3	38.6032
Net Delivery Weight #1	24.8980
Net Delivery Weight #2	24.8878
Net Delivery Weight #3	24.9112
Average Net Delivery Weight	24.8990
Standard Deviation (n-1)	0.012
% Relative Standard Deviation	0.05
Correction factor from Table for EXAX Glass	0.3116
Correction Scale Factor (compared to 100 mL)	0.25
Net Additive Correction (0.25 x 0.3116)	0.0779
Corrected Volume (24.8990 + 0.0779)	24.98

EXAX GLASS

T °C	660	680	700	720	740	760
20.0	0.2725	0.2753	0.2780	0.2808	0.2836	0.2864
20.5	0.2815	0.2843	0.2871	0.2898	0.2926	0.2954
21.0	0.2909	0.2937	0.2965	0.2992	0.3020	0.3048
21.5	0.3005	0.3033	0.3060	0.3088	0.3116	0.3143
22.0	0.3104	0.3132	0.3160	0.3187	0.3215	0.3242
22.5	0.3205	0.3233	0.3260	0.3288	0.3315	0.3343

The sample data come from the table shown. The full tables follow, and cover a wider range of temperature and pressure. The table applies to a volume of 100 mL, requiring that the data be scaled by proportion to the size of the vessel being certified.

Corrections to the Apparent Weight of Water to 20 °C - 100 mL Volume

Source: Care and Handling of Glass Volumetric Apparatus, Kimbal Glass, p. 19; NBS-IR 74-461: The Calibration of Small Volumetric Apparatus.

KIMAX GLASS

T °C	660	680	700	720	740	760	780	800
18.5	0.2446	0.2473	0.2501	0.2529	0.2557	0.2585	0.2613	0.2641
19.0	0.2536	0.2564	0.2592	0.2620	0.2647	0.2675	0.2703	0.2731
19.5	0.2629	0.2657	0.2685	0.2713	0.2740	0.2768	0.2796	0.2824
20.0	0.2725	0.2753	0.2780	0.2808	0.2836	0.2864	0.2692	0.2919
20.5	0.2823	0.2851	0.2879	0.2906	0.2934	0.2962	0.2990	0.3017
21.0	0.2924	0.2952	0.2990	0.3007	0.3035	0.3063	0.3091	0.3118
21.5	0.3028	0.3056	0.3083	0.3111	0.3139	0.3166	0.3194	0.3222
22.0	0.3134	0.3162	0.3190	0.3217	0.3245	0.3272	0.3300	0.3328
22.5	0.3243	0.3271	0.3298	0.3326	0.3353	0.3381	0.3409	0.3436
23.0	0.3354	0.3382	0.3410	0.3437	0.3465	0.3492	0.3520	0.3547
23.5	0.3468	0.3496	0.3523	0.3551	0.3578	0.3606	0.3633	0.3661
24.0	0.3585	0.3612	0.3640	0.3667	0.3695	0.3722	0.3750	0.3777
24.5	0.3704	0.3731	0.3758	0.3786	0.3813	0.3841	0.3868	0.3896
25.0	0.3825	0.3852	0.3880	0.3907	0.3934	0.3962	0.3989	0.4017
25.5	0.3949	0.3976	0.4003	0.4031	0.4058	0.4085	0.4113	0.4140
26.0	0.4075	0.4102	0.4129	0.4157	0.4184	0.4211	0.4239	0.4266
26.5	0.4203	0.4231	0.4258	0.4285	0.4312	0.4340	0.4367	0.4394
27.0	0.4334	0.4361	0.4389	0.4416	0.4443	0.4470	0.4498	0.4525
27.5	0.4467	0.4495	0.4522	0.4549	0.4576	0.4603	0.4631	0.4658
28.0	0.4603	0.4630	0.4657	0.4685	0.4712	0.4739	0.4766	0.4793

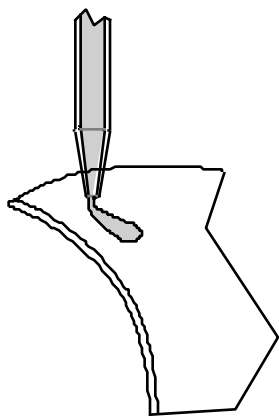
EXAX Glass

(Almost all of the glassware in Chem 256 lab is Exax unless marked Kimax)

T °C	660	680	700	720	740	760	780	800
18.5	0.2423	0.2450	0.2478	0.2506	0.2534	0.2562	0.2590	0.2618
19.0	0.2521	0.2549	0.2576	0.2605	0.2632	0.2660	0.2688	0.2716
19.5	0.2621	0.2649	0.2677	0.2705	0.2732	0.2760	0.2788	0.2816
20.0	0.2725	0.2753	0.2780	0.2808	0.2836	0.2864	0.2892	0.2919
20.5	0.2815	0.2843	0.2871	0.2898	0.2926	0.2954	0.2982	0.3009
21.0	0.2909	0.2937	0.2965	0.2992	0.3020	0.3048	0.3076	0.3103
21.5	0.3005	0.3033	0.3060	0.3088	0.3116	0.3143	0.3171	0.3199
22.0	0.3104	0.3132	0.3160	0.3187	0.3215	0.3242	0.3270	0.3298
22.5	0.3205	0.3233	0.3260	0.3288	0.3315	0.3343	0.3371	0.3398
23.0	0.3309	0.3337	0.3365	0.3392	0.3420	0.3447	0.3475	0.3502
23.5	0.3415	0.3443	0.3470	0.3498	0.3525	0.3553	0.3580	0.3608
24.0	0.3525	0.3552	0.3580	0.3607	0.3635	0.3662	0.3690	0.3717
24.5	0.3636	0.3663	0.3690	0.3718	0.3745	0.3773	0.3800	0.3828
25.0	0.3750	0.3777	0.3805	0.3832	0.3859	0.3886	0.3913	0.3941
25.5	0.3866	0.3893	0.3920	0.3948	0.3975	0.4002	0.4030	0.4057
26.0	0.3984	0.4011	0.4038	0.4066	0.4093	0.4120	0.4148	0.4175
26.5	0.4105	0.4133	0.4160	0.4187	0.4214	0.4242	0.4269	0.4296
27.0	0.4228	0.4255	0.4283	0.4310	0.4337	0.4364	0.4392	0.4419
27.5	0.4354	0.4382	0.4409	0.4436	0.4463	0.4490	0.4518	0.4545
28.0	0.4482	0.4509	0.4536	0.4564	0.4591	0.4618	0.4645	0.4672

Certification of the 25 Milliliter Pipette

1. The pipette should be clean. Verify this. Fill a 400 mL beaker with deionized water. Look through the beaker to make sure that there are no suspended solids in the water.
2. Place a thermometer in the beaker. Record the temperature of the water as the certification is being done. If the water temperature differs from the room temperature by more than a degree, wait to do the certification until thermal equilibrium has been reached.
3. Fill the pipette to at least two inches above the mark. Hold the water level in the pipette for at least a minute to help achieve thermal equilibrium between the water and the glass. Drain the pipette into the sink, observing that it drains a clean film without breaking.
4. Obtain the appropriate "catch bottle" from **Manager, Staff, or Upper Management**, or the stockroom. Shake out any loose material and then cap it. Use the analytical balance to obtain the tare weight of the bottle. Record the weight to **0.000x** grams significance.
5. Fill the pipette at least two inches above the mark with deionized water. Record the temperature of the water in the 400 mL beaker.
6. Using a tissue, wipe the excess water off the external sides of the pipette near the tip. Blot the tip. Do not let water drain from the pipette while doing this.
7. Holding the pipette over a waste beaker, slowly bring the meniscus to the mark and hold it.
This is simpler if the mark is initially at eye level.



8. A small drop usually will remain on the very tip of the pipette. Carefully and slowly move the pipette down to the rim of the waste beaker and touch the tip to the glass. The drop will run off the tip into the beaker. Observe the figure at the left.

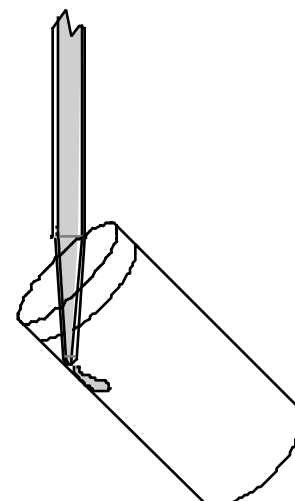
9. The pipette is ready to deliver. Do not bump or jar it.

10. Slowly move the pipette over the opening of the weighed catch bottle. With your free hand, tip the bottle slightly, and touch the tip of the pipette to its inside wall.

11. Holding the pipette vertical, release your finger from its end and allow the water to drain freely into the catch bottle. Be patient and do not shake the setup.

12. When the flow stops, hold the tip in contact with the wall of the catch bottle for at least 60 seconds. Then slowly vertically withdraw it, without bumping it. Observe the figure above.

13. Lay the pipette aside and cap the bottle. Weigh the bottle and record the weight. Check the temperature of the water in the 400 mL beaker. If it differs from your earlier reading by more than 0.5 degree, then record the average of the two readings as the temperature of the water delivered.



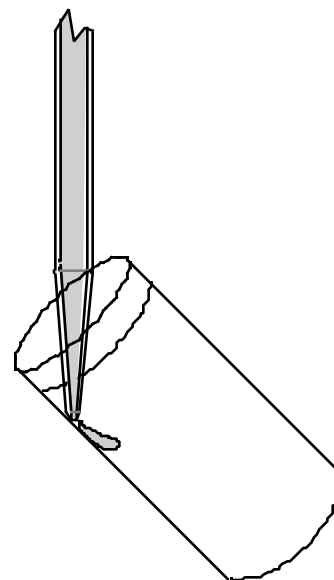
14. Repeat the delivery process as described above. Uncap the catch vessel immediately prior to refilling the pipette. Note that three separate additions are required for three separate weights. **Manager** must choose whether to use 3 separate catch bottles or deliver addition 25 mL portions on top of the first in the same catch vessel.
15. Recap the bottle and weigh it again.
16. Following the sample calculation procedure shown previously, calculate the volume delivered by the pipette.

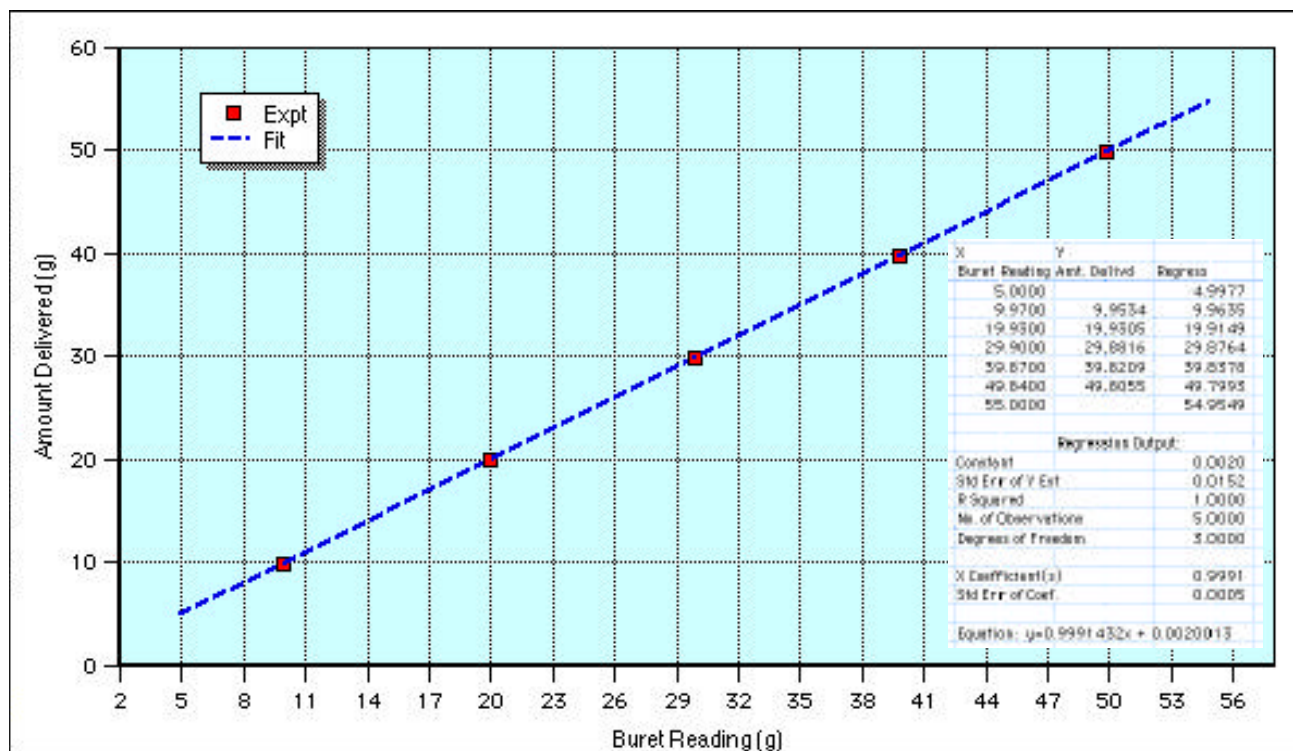
An example of an effective pipette certification is shown below:

DATE: 2/19/93		
Time 14:34		
Manager:	Mark Brommer	
Chemist:	Mark Brommer	
Software:	Jon Falkenberg	
Hardware:	Tim Griffin	
Company:	WenFri	
Item	10 ml. Pipette	25 ml. Pipette
Type of Glass	KIMAX	KIMAX
Capacity in ml.	10	25
Barometric Pressure	734.90	734.90
Temperature(C)	22.20	21.80
Wt. Catch Vessel	28.23	28.23
Delivery Weights		
1	38.18	53.08
2	38.18	53.08
3	38.18	53.08
4	38.18	53.08
Average net wt. A	38.18	53.08
Average Net Del wt.	9.94	24.89
Standard Deviation	0.0085	0.0800
% Relative Standard Dev.	0.0859	0.0800
Correction factor	0.3353	0.3139
Correction scale	0.1000	0.2500
Net add Correction	0.0335	0.0785
Corrected Volume	9.98	24.97
Labelled Volume	10.00	25.00
% Relative Error	-0.25	-0.11

Certification of the 50 Milliliter Burette

1. The burette should be clean and filled with deionized water before starting this part of the experiment. The same catch bottle that was used to certify the pipette should be used here. The bottle should be clean.
2. Dry the bottle inside and out with a tissue. Cap it, and obtain its weight using the top loading balance.
3. Deliver about 25 mL of deionized water from the burette into the catch bottle. Observe that the burette is draining cleanly without spotting. Use the thermometer to measure the temperature of the water delivered.
4. Empty the bottle, dry it with a tissue, cap it, and reweigh it. Unless they differ by more than 0.01 g use the average of the two weights as the tare weight of the bottle, otherwise use the latter reading.
5. Refill the burette to at least an inch above the zero mark. Measure and record the temperature of the water used to fill the burette. Then insert the thermometer into the burette barrel and measure and record the temperature of the water. Use the average of the three temperature readings taken as the temperature for the certification.
6. Wipe the outside of the burette dry with a tissue. Blot the tip. Then drop the liquid level in the burette to about 0.5 mL below the zero mark. An exact zero is neither sought nor particularly desirable.
7. A small drop will likely be left on the tip of the burette. Raise the waste beaker and touch the rim of the beaker to the tip of the burette. The drop should run into the beaker.
8. Record the initial burette reading to 0.0x tolerance. Place the weighed catch bottle below the tip, and tilt it slightly. Raise it until the tip touches the inside wall of the bottle. Observe the figure at the right.
9. Slowly (e.g., at a rate of about 10-15 mL per min.) deliver about 40 mL of water into the bottle. then close the stopcock. Hold the tip of the burette against the inside wall of the bottle for a few seconds, and then remove the bottle by lowering it straight down without bumping the tip. No drop should be on the tip.
10. Cap the bottle and use the top loading balance to determine the weight of water delivered.
11. After a period of no less than 3 minutes, make the final burette reading to 0.0x significance.
12. Following the sample calculation procedure shown below, calculate the volume delivered by the burette.
13. Complete the following measurements:
 - Repeat the certification at more than one volume and construct a curve of apparent vs. measured volume delivered.
 - Determine how much error is introduced in delivering around 40 mL from a burette as a function of how much time is allowed between cessation of delivery, and making the final reading.





An example of an effective burette certification is shown above.

What Constitutes “Round Robin Testing”?

Manager and **Software** have in this experiment the task of setting up a “round robin test” between all of the Companies in the class. This is the only way that we have of determining the accuracy of an analysis when there are no external standards to which test results can be prepared. The concept of a round robin test is to certify one test pipette as having a known value by having the same identical pipette certified by each individual Company. It is important to note that the same pipette is passed between all of the Companies.

In an ideal situation, each Company would devise a procedure for certifying the pipette independently of all of the others, and basically without their knowledge. If we have eight Companies, we would expect to get back, ideally, eight identical answers.

Clearly, if each Company adopts its own way of cleaning, handling, and certifying the pipette, not all of the answers will be identical. There will be both random and determinate error differences between the Companies. But, if all of the differences are within what we determine to be acceptable error bounds, and all of the errors are in fact random, then we can average together all of the values from all of the Companies, and obtain an experimental mean that can be used as a “true” value. With this in hand, any individual Company can then recertify the known pipette to test out their own technique.

The way to interpret the round robin results is to use a t-test. The one described in Harris is too laborious to do in a lab situation like this. Instead, we will use a Excel tool that will calculate the probability that two mean values are the same.

Consider the following table of results on a 25 mL pipette certification; although the same pipette was certified by several (but unfortunately not all) of the Companies, the average values do in fact differ from each other. We are concerned. Looking at any Companies, we wonder if the differences are significant? In other words, do the average values differ from each other because the spread of results was large, or do they differ because there was some systematic or determinate error at work?

Another way of saying this, a bit easier to understand is, what is the probability that two average values are significantly indistinguishable from each other? If it was 95% probable that they were indistinguishable, and had different numerical values only because of the standard deviations in their determinations, then we would say that they are “statistically equivalent” at the 95% confidence level. We strictly cannot say this. The t-test may not be powerful enough to distinguish between means, and we may have too small a sample. But, for the use of this particular lab it will be OK to do so.

25-mL pipette (EXAX)			
Company	Volume	STD.	# of trials
wentue	24.8326	0.0042	4
lautue	24.8521	0.0156	4
brutue	24.7919	0.0133	5
deatue	24.9871	0.0104	4
wenwed			
lauwed	24.8472	0.0618	3
bruwed			
deawed			
wenfri			
laufri	24.8833	0.0132	4
brufri	24.9874	0.0198	5
deafri			

Although the same pipette was certified by several (but

Is this good enough? We will agree that if 95 times out of 100 the way in which the pipettes were certified would produce a statistically equivalent result, then the numbers are the same. Clearly there is a lot of testing possible in the above table. The approach that we have used in the past is to t-test two values against each other. If they are not distinguishable at the 95% level, then, add their variances and take the square root of their sum to use as a new standard deviation.