MEASURING ERRORS
Calibrating and using volumetric glassware

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English

Summary
The *Grand Prix Chimique* is a European contest for vocational chemistry. Could you represent your country? To be sent to this competition you must be among the best. This is determined by seeing how well you perform analyses and find true values.
In this activity, two groups of students compete in a virtual analytical competition. One group will use calibration and another group will just use the glassware as it is. Is there any difference? Does the use of the actual volumes of pipettes, burettes and flasks get you closer to the final of the Grand Prix Chimique?

Activity type **A**
Use of scientific knowledge and understanding to solve problems
Working in teams to solve problems

Techniques
Complexometric titration
Calibration of glassware

Field
Water quality
Calibration

Time
Practical lessons: 135 minutes
Theory lessons: 75 minutes
Out of class time: 60 minutes

StandardBase procedures
Calibration of glassware
Calibration of digital burette

Determination of hardness of water using complexometric titration

StandardBase techniques
Volumetric and gravimetric analysis
Complexometric titration

Other resources
Brand Glassware: www.brand.de
www.iupac.org/publications/pac/1994/pdf/6612x2527.pdf
http://www.gpch.hu/history.htm
http://standardbase.vapronet.nl/userdata/sbase_beheer/documenten/stappenplannen/Determination_of_hardness_of_water_(Step-By-Step_Plan).pdf
http://www. standardbase.com
Others are given within the activity.

Student's document

MEASURING ERRORS: Calibrating and using volumetric glassware

The European Grand Prix Chimique is a competition between vocational students in chemistry. It is held in every two years. In each participating country, there is a preliminary national Grand Prix Chimique competition. In each national Grand Prix Chimique, the best students also compete in analytical chemistry. The best three students from each country are sent to the European finals. For the European finals of the Grand Prix Chimique, the jury consists of experts from schools and industry. The jury looks at performance of skills and practical work. But how can they determine who is the best? The Grand Prix Chimique jury makes a list of skills that need to be judged (filling up volumetric flasks, pipetting, determining the endpoint of titration by colour etc.). Besides judging skills, analytical results are also taken into consideration. So, the best three will most likely be the three students whose average of their measurements is closest to the true value. The jury also considers the smallest standard deviation.

In this project, you have to find out if the apparatus you use has any influence on the results. Is any spread in results caused by poor, inaccurate glassware? Does that really make a difference?

Students are divided into two groups. One group makes all the necessary calibrations of pipette, flasks and burette before, or after, titration. When these are calibrated, you will know whether the 100 mL has in actual fact become 100.5 mL over a period of time. Another group takes the glassware as it is. Does the use of the actual volumes of pipettes, burettes and flasks get you closer to the final of the Grand Prix Chimique?

Your brief

The determination of hardness of water by complexometric analysis is carried out by at least two groups, using a control sample or commercial calcium solution as the 'water sample'. One group chooses pipette, volumetric flasks and burette and whatever else is needed, without considering glassware class. The other group must calibrate all the glassware used (before or after titration) and use these values to recalculate results. You will compare the results of both groups and give an answer to the question: "Does calibration of glassware really improve the results for this titration?"

Your investigation

All groups

Task 1

Look up the suggested *StandardBase* procedures.

Form two groups: one is called the "Grand Prix Chimique group", the other the "Fast and furious group". The "Grand Prix Chimique group" should have three times as many students as the "Fast and furious group" (six and two, for example).

- Make sure all group members understand the goals.
- Each group will have its own meeting to divide workload among members.
- Appoint a project leader/chairman, secretary and a collector of all results.
- Estimate the time needed for all activities (see the *StandardBase* website).

- What number of analyses seems likely to be needed?
- Consult the other group, and agree the number of analyses to carry out.

Fast and furious group

Task 2

- Use www.standardbase.com *Determination of hardness of water using complexometric titration.*
- Perform the analyses.

Grand Prix Chimique Group

Task 2

- Use the **student sheets** at the end of this document to calibrate the glassware.
- Use www.standardbase.com *Determination of hardness of water using complexometric titration* (when necessary look at *Calibration of glassware).*

Calibration can be done in parallel with the actual analyses. It is also possible to calibrate the glassware afterwards and recalculate results using actual volumes.

- Perform the analyses.

Both groups

Task 3

Report to the other group by giving mean and standard deviation of both EDTA concentration and calcium ion content [as $c(Ca^{2+})$ in mol/L] in the sample. Check the number of analyses. Compare and evaluate the results/outcomes of both groups. Comment the outcome: who is the best, based on what proof? Was the calibration really necessary for better results?

Notes

A risk assessment must be carried out before starting practical work.
Calibration of glassware can be mixed with the titration itself. Be sure to mark your glassware for later use.
Discuss the plan with your teacher (how many sample titrations do you think you can carry out?), including instruments, glassware and chemicals.

Data sheets and tips for independent search for information

http://www.cofc.edu/~kinard/221LCHEM/CHEM221L%20Calcium%20Determination%20by%20EDTA%20Titration.htm
http://www.brand.de
http://en.wikipedia.org/wiki/Hard_water
http://www.chemistry.wustl.edu/~edudev/LabTutorials/Water/FreshWater/hardness.html
http://www.water-research.net/hardness.htm

Your findings

Write a short report with your results and conclusions; provide a summary of results (mean and standard deviation) and judge how well your group performed. Compare your group's result with that of the other group. On what basis would you choose your strategy to win the Grand Prix Chimique?
Exchange and compare the results from all groups. What is the confidence level of your findings?

Student self assessment

Use the file *Evaluating skills* from the ProBase website.

Student sheet: Calibration of volumetric glassware

Calibration of volumetric equipment is carried out by weighing the amount of water contained or delivered. Since the density (or mass per unit volume) of water changes, the temperature of the water used in the calibration must be known. The actual volume delivered can be obtained from a table relating temperature to volume, such as the table below.

Volume occupied by one gram of water (specific volume) at various temperatures, corrected for buoyancy

Temperature (°C)	Volume (mL)	Temperature (°C)	Specific volume (mL/g)
18	1.0025	26	1.0043
20	1.0029	27	1.0046
22	1.0033	28	1.0048
24	1.0038	29	1.0051
25	1.0040	30	1.0054

Although errors in burette, pipette and volumetric flask markings by the manufacturer are usually small, they cannot be neglected in accurate work. For example, if a 10 mL pipette actually delivers 9.99 mL, an error of one part per thousand will be introduced unless the true value is employed.

Example for a 10 mL pipette

Temperature of water	26 ^{O}C
Mass of flask and water	24.688 g
Mass of flask	14.713 g
Apparent mass of water delivered	9.975 g
True volume of pipette	10.02 mL

At 26 ^{O}C, 1 g of water occupies 1.0043 mL.
The true volume of 9.975 g of water at 26 ^{O}C is

$$9.975 \text{ g} \times 1.0043 \text{ mL/g} = 10.02 \text{ mL}$$

Use this corrected volume in calculations for titrations.

Student sheet: Example datasheet

Name: _____ Date: _____

CALIBRATION OF THE 25 mL PIPETTE

Water temperature _____

_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL

Mean: _____ mL
Standard deviation: _____ mL

BURETTE CALIBRATION

Water temperature _____

10 mL

_____ g _____ mL
_____ g _____ mL
_____ g _____ mL

Mean: _____mL
Correction factor _____
Standard deviation: _____mL

20 mL

_____ g _____ mL
_____ g _____ mL
_____ g _____ mL

Mean: _____mL
Correction factor _____
Standard deviation: _____mL

30 mL

_____ g _____ mL
_____ g _____ mL
_____ g _____ mL

Mean: _____mL
Correction factor _____
Standard deviation: _____mL

40 mL

_____ g _____ mL
_____ g _____ mL
_____ g _____ mL

Mean: _____mL
Correction factor _____
Standard deviation: _____mL

50 mL

_____ g _____ mL
_____ g _____ mL
_____ g _____ mL

Mean: _____mL
Correction factor (CF) _____
Standard deviation: _____mL

Draw a graph of *volume indicated* versus *correction factor* using the mean value from your calibration to determine the correction factor.
[CF = (volume delivered) - (volume indicated)]
Attach the graph to your report.

VOLUMETRIC FLASK CALIBRATION

The calibration of volumetric glassware is performed in a similar way as for pipettes and burettes. The basic idea is to weigh the flask empty and when filled. Calculate the difference in mass and then find the volume using the density of water at the relevant temperature. Make sure the flask is dried inside with a little acetone/water and then blown dry using a hairdryer (without heating). Calculations are similar to those given above.

100 mL volumetric flask

Water temperature _____

_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL
_____ g _____ mL

Mean: _____mL
Standard deviation: _____mL

Teacher's document

Overview

Students learn about the advantages and disadvantages of calibration of laboratory apparatus, and learn how systematic errors can be minimised. They choose the best method for the determination.

Developing students' skills

All the students will carry out a complexometric titration, some with and others without calibration. They should receive feedback after each one. Appropriate questions should be asked as prompts to think about.

The development of skills will depend on the required assessment objectives for the course being followed by the students. The completed report on the 'best' method for the determination of the complexometric titration may all provide summative assessment evidence, judged against published grading criteria.

Organising and managing the class

Minimum group size:
"Grand Prix Chimique group": 6; "Fast and furious group": 2.
The first group can organise calibration and titration in parallel. Half of the calibrations could be done after titration. Volumes must be recalculated in whichever case. The "Grand Prix Chimique group" uses actual volumes.

Number of groups: at least 2.
One group uses all possible calibrations; the second group takes the easier way. The two groups should do the same number of analyses. This requires estimation of the length of each practical activity.

This project structure can be used on any type of titrimetric analysis (acid-base, redox etc). You can use different procedures as long as there are two rival groups.
In a larger class (16 or 24 students) you could have each analysis performed by the 2 groups. So, with 24 students, three different titrimetric analyses are possible.

A valuable way of enhancing the groupwork is for the students to go around the class, judging the skills of their peers. They should make a list of what they will scrutinise.

Session plan for teachers

A suggested approach is:

- Teacher's introduction to the scenario (1 x 30 minutes)
  - outline the available equipment and chemicals
  - statement of expected output (content, format)

Give an example where the 100 mL volumetric flask appear to be 101 mL and the pipette of 10 mL is actually 9.91 mL

- Collecting information and diagnostic assessment with questions related to the subject (1 x 45 minutes)
- Carrying out practical work (3 x 45 minutes)
- Making the final report, evaluating results (1 x 60 minutes)

Technical notes

General remark

This project can be done using any analyses that make use of calibrated glassware. The calcium titration could just as well be replaced by any straightforward analysis. With a larger number of students, you could additionally have an AAS determination of iron in the same way. There should always be two groups competing with the same analyses.

▪ Equipment and materials

Glassware

The actual quantity of each of the following depends on the number of students.

- 100 mL volumetric flasks (Class A)
- 20 mL pipette (Class A)
- 50 mL burette
- conical flasks for titration
- analytical balance
- 50 mL beaker
- conical flask with grinded stopper (for calibration with water)
- weighing bottle (for solids)

Chemicals with their CAS numbers

ammonium chloride	NH_4Cl	12125-02-9
ammonia solution		7664-41-7
w = 25%, ρ (20 °C) = 0.91 g/mL		
disodium magnesium salt of EDTA	$C_{10}H_{12}N_2O_8Na_2Mg$	14402-88-1
disodium salt of EDTA dehydrate	$C_{10}H_{14}N_2O_8Na_2.H_2O$	6381-92-6
mordant black 11	$C_{20}H_{12}N_3O_7SNa$	1787-61-7
eriochrome black T		
hydrochloric acid, 4 mol/L		7647-01-0
methyl red indicator		493-52-7
triethanolamine	$(OHCH_2CH_2)_3N$	102-71-6
ethanol		64-17-5
acetone		67-64-1
calcium carbonate		471-34-1
calcium chloride		10043-52-4

▪ Preparation

Solutions

- Prepare a 0.01 mol/L EDTA solution.

See 3.4.2 in the *StandardBase* procedure

- Prepare a calcium standard reference solution, 10 mmol/L

See 3.4.3 in the *StandardBase* procedure.

Give the exact concentration of the solution on the label.

- Use this solution for standardisation of the 0.01 mol/L EDTA solution

Standardisation EDTA solution: solution calcium carbonate - see the procedures on the *StandardBase* website.

Make sure both groups have the same EDTA solution and the same sample. Before starting the titration, make sure the pH of the calcium solution is approximately 10. A lower pH obstructs the formation of the Ca-EDTA complex.

Preparation of the calcium chloride control sample
It is recommended to use a technician-made sample, to be able to compare the average results of the groups. This can be prepared in the same way as the standard calcium solution is prepared.
Your own control sample or commercial calcium solution will also do.

Weigh accurately about 110.0 mg of calcium chloride in a weighing bottle. Quantitatively transfer the weighed calcium chloride to a 50 mL beaker and dissolve the calcium chloride.
Transfer this solution to a 100 mL volumetric flask. Rinse the beaker, two or three times, with distilled water and add the washings to the volumetric flask. Add more distilled water to the flask until the solution is about one centimetre from the graduation mark. Use a dropping pipette to make up to the mark with distilled water. Stopper the flask and invert several times to ensure that the contents are thoroughly mixed. Pipette 20 mL of this solution into a conical flask. Hereafter, follow the procedure for the determination of hardness of water from the *StandardBase* website.

The students should not know the exact concentration. You can give an estimate close to the expected value.

Note
The Standard Operating Procedure (SOP) procedure to calibrate Brand TC glassware (volumetric flask) is based on ISO 4787. According to this standard, the calibration procedure for volumetric flasks differs from normal straightforward procedures used in analytical laboratories. Feel free to use this method, but add an explanation that the method of adjusting to mark must not be used in the case of preparation of solutions (dilutions).

TESTING PRIOR KNOWLEDGE

Choose the best answer

1. The reaction between Ca^{2+} ions to EDTA in molar ratio is
   a) 2 :1
   b) 1 :1
   c) 1 :2
   d) unknown

2. What happens when the pH is above 11?
   a) The reaction rate will be decreased slightly
   b) Nothing important changes
   c) CO_2 gas will be absorbed from the air
   d) The Ca-EDTA complex is not stable enough

3. Before actual end results of both groups are known you expect the "Grand Prix Chimique" results compared with the "Fast and furious" results to:
   a) have a larger standard deviation
   b) have a smaller standard deviation
   c) have higher mean values
   d) have lower mean values

Note: answers are given in red. These need to be removed before the test is used.

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