Two applications of gravimetric titration: simple, buret-free, and high precision

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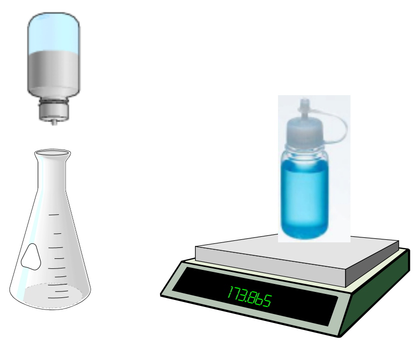
Introduction

Here are two example applications of gravimetric titration that are suitable for laboratory experiments in senior secondary school chemistry courses or in college general chemistry courses. This material is excerpted or adapted from two articles (1,2) that may be downloaded from my web pages.

Gravimetric Titration

Gravimetric titration with a 60 mL polymer controlled drop-dispensing squeeze-bottle and a 2-place digital balance is simpler, faster, less costly, and more precise than titration with a 50 mL buret (**Graphic 1**). A complete description of the method, its advantages, and the equipment used are found in reference 1.



Graphic 1: Gravimetric Titration with a Polymer Controlled Drop-Dispensing Squeeze-Bottle and a 2-Place Digital Balance

Acid Content of Fruit Juices and Soft Drinks

The total acid content of a fruit juice or a soft drink is easily determined by gravimetric titration with 0.1 M NaOH solution, either using phenolphthalein as indicator or a pH meter to pH > 8 as the end-point. Some typical titration results for titration of acids in fruit juices and soft drinks are found in **Table 1**. The titration mass values stated are approximate, because the 0.1 M NaOH solutions used were not standardized. All of the results are the means of at least two titrations. The major acid present is noted in each case. Full details of this experiment and student instructions are found in reference 1.

Table 1: Titrations of 10 mL Samples With 0.1 M NaOH Solution

Sample	Titration Mass (g)
Kool-Aid® Invisible (Citric)	4.31
Welch's® White Grape Juice (Tartaric)	6.35
Sun-Rype® Apple Juice (Malic)	6.30
Sprite®** (Citric)	2.50
Canada Dry® Ginger Ale** (Citric)	2.45
Coca-Cola®** (Phosphoric)	1.50*
Black River® Purple Grape Juice (Tartaric)	10.00*
Black River® Cranberry Juice (Succinic)	> 15*

* pH Meter Titrations to > pH 8 ** Carbonated Drinks Degassed

What Precision Can Students Achieve on a Gravimetric Titration Experiment?

Michael Jansen of Crescent School in Toronto kindly invited me to his classroom in February 2012, to try the experiment with a group of eight grade 11 chemistry students who participated in a weekly extracurricular chemistry session. The group (Owen, Carter, Ryan, Jonathan, Nick, Kevin, Scott, and Jorgen) worked as pairs to analyse Tim Hortons® and SunRype® apple juices, which proved to be very similar in acid content.

In an 80-minute period, without any advance preparation, the students performed some rough trials and then 5 or 6 analyses per group. The analysis of 5 mL samples of juice, measured by graduated cylinder, was performed by both drop count and gravimetrically, using a 2-place digital balance. Drop count is used as an aid when doing replicate titrations of the same sample solution. The best set of results, from one pair, is listed in **Table 2**. The abbreviation CI stands for confidence interval. The value of the 95 % confidence interval of the data set was obtained using the statistics engine at the website WolframAlpha (3).

The students obtained very good precision on a ≈ 2.7 g titration, as indicated in the table. The range of the 95 % confidence interval was ≈ 3.4 % of the mean value of the titration.

Table 2: Gravimetric Titrations of 5 mL Samples of Tim Hortons Apple Juice with 0.1 M NaOH		
Trial	**Mass (g)**	**Drops**
1	2.77	72
2	2.60	72
3	2.66	74
4	2.64	74
5	2.67	75
6	2.69	74
Mean	2.67	
95 % CI	2.63 - 2.72	
Range of 95 % CI	0.09 (± 0.01)	
Range of 95 % CI as % of Mean	3.4 (± 0.4) %	

Comparing the Precision of the Gravimetric and the Volumetric Methods

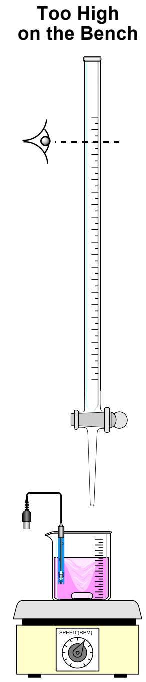
In order to compare the relative precision of the gravimetric method and the volumetric method of titration, both methods were used to standardize a 0.1 M NaOH solution. Samples of the primary standard substance potassium hydrogen phthalate (KHP) were weighed by 4-place analytical balance, and titrated with the 0.1 M NaOH solution using phenolphthalein indicator.

The results of this experiment indicated that the gravimetric method is more precise than the volumetric method. The gravimetric method needs just less than half the sample amount to give the same precision as the volumetric method. The full results are described in reference 2.

Gravimetric pH Titration Curves

The gravimetric titration method is well suited to the manual development of a pH or other potentiometric titration curve. Two important factors here are the physical simplicity of the method, and the ease with which the titrant solution may be metered into the titration vessel. A concrete description, of a before and after case from the second term analytical chemistry laboratory for the two-year technician program at Mohawk College, will be used to illustrate these points. This is described fully in reference 2.

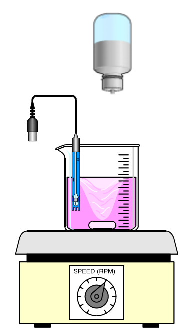
Previously, the students were asked to use the volumetric method to record the titration curve of a solution containing an unknown concentration of carbonate and bicarbonate ion. Although they were familiar with the use of a stirring motor and PTFE-coated stirring-bar, a combination electrode, and a pH meter, they had difficulty putting the 50 mL volumetric buret in place over the titration beaker (**Graphic 2**).



Graphic 2: Volumetric pH Titration

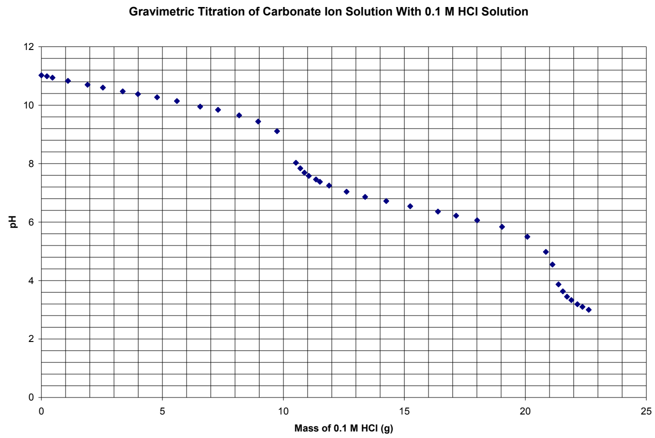
They then had great difficulty getting up high enough over the bench to read the level in the buret, and found it nearly impossible to control the small additions of titrant desired for the titration curve measurements. The exercise was overlong for a 2 hour period; it was hazardous because the students were climbing up on the bench to read the buret; and the titration curves produced were of poor quality.

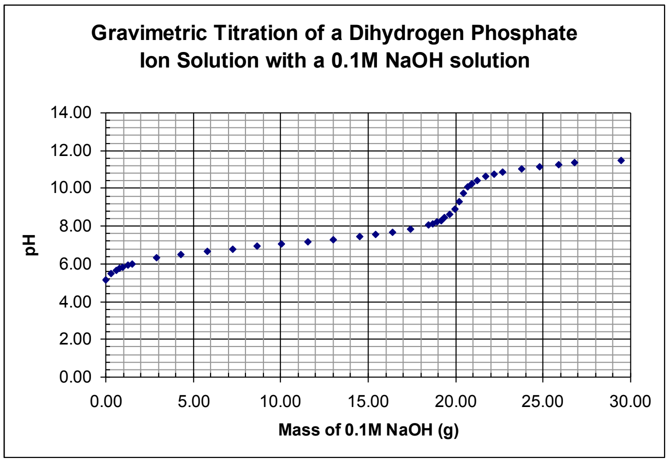
Switching to the gravimetric titration method solved these problems. Using a polymer drop-dispensing squeeze-bottle instead of a buret allowed the titrant solution to be easily metered directly into the beaker by counting drops (**Graphic 3**). The mass of the added reagent was determined by measuring the mass of the squeeze-bottle after each addition on a two-place top-loading balance. When the pH was changing slowly, more drops (e.g. 30 drops), and when the pH was changing rapidly, less drops (e.g. 5 drops) were added between successive pairs of pH - mass measurements.



Graphic 3: Gravimetric pH Titration

It was found that in the same 2 hours of laboratory time, two different pH titrations curves of good quality could be determined, as illustrated by the plots shown. The full experiment script can be downloaded from my web pages (4).





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