High Precision Titrations Without a Buret: Acid Content of a Fruit Juice or a Soft Drink

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Summary

Here is a **simple**, **quick**, and **low cost**, yet **high precision** titration analysis experiment for senior chemistry students. It is a quality-control-type analysis to determine the amount of acid present in a fruit juice or a soft drink, by titration of samples with dilute sodium hydroxide solution from a **controlled drop-dispensing polymer squeeze-bottle** using phenolphthalein indicator. A polymer dropper squeeze-bottle is far less costly than a buret and is much simpler to use. The experimental measurement is the mass of titrant solution used, determined using a 2-place digital balance. The chemical solutions required are low cost, relatively safe for use, and non-hazardous for disposal.

Contents of the Article

1. Introduction

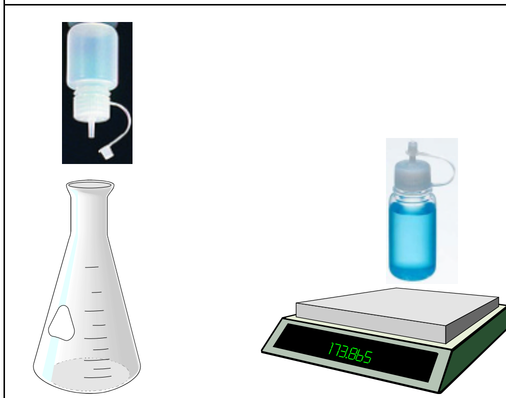
Fruit juices are acidic, containing mixtures of the weakly acidic **fruit acids**: the predominant acid is **citric** acid in citrus juices; **tartaric** acid in grape juices (and wines); **malic** acid in apple juices; and **succinic** acid in cranberry juices (1). Synthetic soft drinks are also made acidic: by **citric** acid in Kool-Aid®, lemon-limes and ginger ales; by **phosphoric** acid in colas.

A commonly used quality control procedure to determine the total amount of acid present in juices or drinks is a weak acid-strong base titration analysis. The titrating reagent used is a **0.1 M sodium hydroxide (NaOH)** solution, which contains 0.4 g of NaOH per 100 mL of solution. For titration of juices, or drinks, which are clear and colourless (or pale yellow), **phenolphthalein** is an excellent acid-base indicator substance.

Traditional student volumetric analysis employs a 50 mL buret. This is an expensive piece of precision glassware, which is difficult to learn to use, easy to break, and requires a great deal of class laboratory time to clean, fill, refill, use properly, empty, and clean out.

A low cost controlled drop-dispensing 60 mL polymer squeeze-bottle, along with a 2-place digital balance may be used (**Graphic 1**) in place of the 50 mL buret. This method, called a **gravimetric titration**, is **simpler**, **faster**, and **less costly**. Students can learn to use a dropper bottle in minutes. The dropper bottles may be filled from a wash bottle in advance of the class, a time saving.

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



The most expensive 60 mL dropper bottles cost only $ 6 each compared to $ 107 each for a 50 mL buret (2). The polymer bottles containing the 0.1 M NaOH solution may be refilled rapidly from the wash bottle during the class as needed, and both the 0.1 M NaOH solution and the phenolphthalein indicator solution may be left in polymer dropper bottles for prolonged periods; there is no need to empty or clean out the bottles. At Mohawk College these reagents remain in the bottles from year to year.

The technique of **gravimetric titration** is described in textbooks of analytical chemistry (3). Gravimetric titrations with a controlled drop-dispensing 60 mL squeeze-bottle and a top-loading 2-place digital balance are more precise than volumetric titrations with a 50 mL buret. For the same precision as the buret experiment, the sample volumes and titration volumes may be less than half as much (4). There is no need to initially fill and finally empty the student burets for each day's experiment, so the amount of titrating reagent solution required is much reduced. The incidence of spills or accidents will be lessened.

2. Experimental Method

The student exercise consists of two parts:

1. Measurement of the required portions of the sample into titration vessels, and addition to each sample of drops of phenolphthalein indicator and distilled water.
2. Titration of each sample with drops of the 0.1 M NaOH solution from the polymer squeeze bottle to the colour change, and measurement and recording of the mass of the reagent dropper bottle before and after each titration.

Teacher Preparation

Possible juices or drinks to use as samples, and suggested sample volumes are given in **Section 3** below. An appropriate sample volume for the method is typically in the range of 5 mL to 15 mL of a juice or a drink. Carbonated drinks must be degassed, to remove the acidic carbon dioxide before titration. Do sample trials with your juice(s) and / or drink(s) to become familiar with the method and to determine the appropriate sample volume(s) for the student exercises.

Label and fill the dropper bottles with the 0.1 M NaOH solution and the phenolphthalein solution.
Chemicals and Safety are discussed in **Section 4** below, and **Equipment, Materials, and Supplies** are discussed in **Section 5** below. The students should be made familiar with their safety instructions and materials disposal instructions before beginning the exercise.

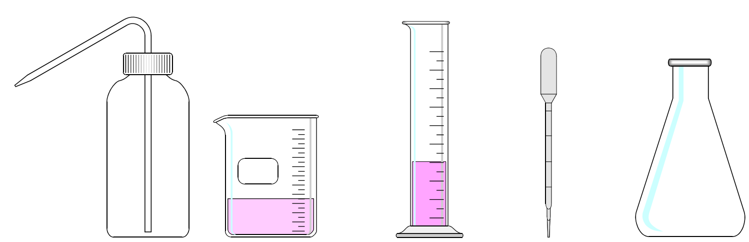
Students should be placed in pairs or groups. They can rotate amongst the various tasks of sample preparation, titrating, and recording the drop counts and bottle masses before and after each titration. Student titrations should be less than 100 drops, or roughly in the range of 2 g to 4 g of the 0.1 M NaOH solution being required; larger titrations will be tedious, will take too long, and are not necessary to achieve good precision.

Student Instructions

WEAR SAFETY GLASSES OR GOGGLES AT ALL TIMES

1. Measure out one or more samples of a juice or drink as instructed, using a small pipet to adjust the level of the sample in the graduated cylinder (**Graphic 2**). Place each sample into a 125 mL Erlenmeyer flask. To each sample, add 4 or 5 drops of phenolphthalein indicator solution and enough distilled or deionized water to reach a total volume of 15 to 20 mL. Mix well.

Graphic 2: Dispense Sample Portions with a Beaker or a Wash Bottle; Adjust the Level in a Graduated Cylinder with a Polymer Pipet; Transfer to an Erlenmeyer Flask for Titration



2. The solutions should be clear and colourless or pale yellow, depending on the sample used. One sample is kept as an un-titrated colour comparison. Use a white or light coloured background for the titration (paper towels could be used), in a well-lit area.

3. Take one of the bottles of the 0.1 M NaOH solution provided. The outside of the bottle and your hands should be clean and dry (**Note 1**). Zero the top-loading 2-place digital balance. Place the bottle on the balance. Record the starting mass to 2 places after the decimal point, using a table format or a supplied data sheet.

Note 1: Clean, Dry Hands
Experience has shown that clean, dry hands will not change the measured mass of the bottle to within ± 0.01 g.

4. Titrate one sample by adding drops of the 0.1 M NaOH solution to the sample flask, swirling the solution periodically to mix well (**Graphic 1**). Hold the bottle vertically (**Note 2**). **Count drops**. The pink colour of phenolphthalein will appear but disappear on mixing. Continue titrating, until the pale pink colour (pale orange for yellow samples) is seen to last for at least several seconds. At this point, if you have a wash bottle filled with distilled water, wash down the inside walls of the flask. Add drops of the 0.1 M NaOH solution until the colour change lasts for at least a full minute or becomes permanent. The solution is now alkaline, and the titration is ended. Record the drop count.

Note 2: Hold the Bottle Vertically
Drop size varies considerably with the angle of the bottle. In trials, measured drop mass was greatest when the bottle was vertical. This means that the bottle must be held in the same position during titrations for consistency of drop counts. For this reason, it is suggested that the bottle always be held vertically during titrations.

5. Zero the top-loading 2-place digital balance. Place the bottle on the balance. Record the final mass to 2 places after the decimal point as before.

6. Titrate a number of further samples of the same or different juices or drinks as instructed. The drop count of repeated titrations of the same sample type should be very similar (**Note 3**). **Discard all waste solutions into a sink or a disposal container as instructed**.

Note 3: Titrating to the Same Drop Count
Since the juices and drinks are homogeneous solutions of uniform composition, repeated titrations of the same sample size are expected to give the same result, except for experimental variation. Repeated titrations of the same juice or drink can be done very rapidly after some practice.

What Precision Can Students Achieve on this 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. The best set of results, from one pair, is listed in **Table 1** (**Note 4**). The students obtained very good precision, as indicated in the table. The **% RSD** value is the standard deviation of the results as a percentage of the mean value; the **95 % CI Range** values give the 95 % confidence interval (5).

Note 4: Results are Approximate
The values obtained in these titrations are an approximate guide only to the analysis, since the 0.1 M NaOH solution used was not standardized.

Table 1: 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	
% RSD	2.1	
95 % CI Range	2.63 - 2.72	

3. Sample Choice and Amount

Any clear, colourless or pale yellow juice or drink is a suitable sample for this method of analysis. Examples include: Kool-Aid® Invisible; Sprite®; Canada Dry® Ginger Ale; any apple juice or white grape juice. Carbonated drinks must be degassed to remove the acidic carbon dioxide before titration. The end-point of titration is indicated by the phenolphthalein as a lasting appearance of a pink or an orange colour, clearly different from an un-titrated comparison sample. Strongly coloured samples can be titrated only with a pH meter. Synthetic drinks are expected to have consistent acid content from sample to sample. Fruit juices, on the other hand, may vary by brand and seasonally.

The results in **Table 2** were obtained in each case as the mean of three or more repeats of 10 mL samples titrated by the gravimetric method, measuring and recording the dropper bottle mass before and after the titration in each case using a 2-place digital balance. These results are approximate (see **Note 4** above).

Table 2: Titrations of 10 mL Samples With 0.1 M NaOH Solution

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

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

Choosing the Sample Amount

The titration mass for these titrations depends on the sample and the sample volume taken. A titration mass of about 2.5 g will be adequate for acceptable precision. Larger titration masses require more time, are more tedious, and reduce the number of titrations the students can do.

As described in **Section 5** below, drop counts vary, depending on the type of bottle used, from about 15 drops per g to 30 drops per g. Students will find it very tiring physically and mentally to do high drop-count titrations.

4. Chemicals and Safety

The two required chemicals are relatively inexpensive, and may be conveniently purchased in the form of dilute solutions in water:

- 0.1 molar NaOH (0.4 g / 100 mL) (**$7.56 / L**) (6);
- 0.5 % phenolphthalein (**$8.83 / 500 mL**) (7).

Sodium Hydroxide

Sodium hydroxide, also called caustic soda or lye, is an important industrial chemical (8) (see **Box**).

Box: Major Uses of Sodium Hydroxide
- Organic and inorganic chemical production; - Paper production; - Natural soap and synthetic anionic detersive production; - Aluminum production; - Industrial acid neutralization.

For industrial and large-scale use, sodium hydroxide is produced and shipped as solid pellets or flakes or as a very concentrated aqueous solution (40 or 50 % by mass). These solutions are also used in the Kjeldahl nitrogen analysis (9). Solid NaOH is very hygroscopic, absorbing water vapour from the air. It also absorbs carbon dioxide from the air.

The heat of solution of NaOH is very large; dissolving the pellets in water or diluting a concentrated solution creates a large amount of heat energy that may boil the solution. Both as a solid and as a concentrated aqueous solution it is an extremely corrosive and hazardous chemical. Concentrated solutions are able to attack human tissue and most kinds of glass and plastic. Despite this, it is sold to consumers in drain cleaner and oven cleaner products (10).

The MSDS for sodium hydroxide is intended for industrial producers and users. It is rather frightening (11). However, dilute solutions of sodium hydroxide such as the 0.1 molar solution (0.4 g / 100 mL) used in this exercise are far less hazardous to use. These solutions are ubiquitous in industrial and academic laboratories, as well as in teaching laboratories at most levels. A 0.1 M solution of sodium hydroxide is not considered hazardous, but it must be respected (see **Table 3** below).

Table 3: Hazards of Using 0.1 M Sodium Hydroxide and 0.5 % Phenolphthalein Solutions	
Hazard	**Remedy / Action**
Eyes	**WEAR SAFETY GLASSES OR GOGGLES AT ALL TIMES** **If an accident does occur, rinse at an eye-wash station according to the instructions.**
Skin	**Rinse off with tap water or wipe off with a dampened cloth. Rinse the cloth under the cold water tap.**
Clothing	**Rinse the affected area with cold water tap.**
Bench or Floor	**Wipe up with a dampened cloth. Rinse the cloth under the cold water tap.**

The total amount of solution required for a class of 30 students, assuming they work as 15 pairs, is unlikely to exceed 2 L of the 0.1 M NaOH. This means the total amount of sodium hydroxide being used will be less than 8 g! The danger to skin or clothing is very minor. Skin will not be affected for many minutes. The only serious hazard is to the eyes.

Students must wear safety glasses or goggles at all times, and must wash their hands before taking the eyewear off. An eye-wash station or eye-wash kit must be at hand during the experiment. Unless you have eye-wash plumbing, a laboratory safety eye-wash solution kit is needed. These are available at a reasonable cost (12), but if you have very limited means, consider purchasing an OTC drugstore eye-wash solution kit (13).

Phenolphthalein
This is one of the most commonly used acid-base indicator substances, typically used in the form of a 0.5 % solution. It has an interesting history (14). The amount actually used in this exercise is very small and it is not considered hazardous in this usage. Use the same precautions as for the sodium hydroxide.

5. Equipment, Materials and Supplies

Table 4: Required Equipment, Materials and Supplies	
Item	**Labware**
Eye Safety	**Safety Glasses or Goggles. Eye-Wash Plumbing or An Eye-Wash Kit**
Titration Vessels	**125 ml Erlenmeyer Flasks**
Sample Dispensing and Measuring Equipment	**Wash Bottles or Small Beakers, Graduated Cylinders and Disposable Pipets (15),** **2-Place Top-Loading Digital Balance**
Controlled Drop-Dispensing Polymer Squeeze Bottles	**60 mL Bottles (NaOH)** **15 mL or 30 mL Bottles (Phenolphthalein)**
Water	**Distilled or Deionized Water (Supermarket)**
Miscellaneous	**Paper Towels (As a Pale Background for Titrations); Wipe-Up Cloths; Wash Bottles**

Dropper Bottles
Three types of dropper bottles suitable for gravimetric titrations are described in **Table 5**. Be very wary of purchasing any other bottles, despite the catalog descriptions. **The key property required is that the drops are dispensed only when the bottle is firmly squeezed**. Larger drops make each titration faster at the cost of end-point precision. An integrated tip / cap combination makes refilling easy, but storage less secure. A snap-out inner tip / closed outer cap combination makes year-to-year storage more secure, at the cost of making the bottle much harder to refill.

Table 5: Polymer Controlled Drop-Dispensing Squeeze-Bottles (see Photograph)			
Bottle Type	**Supplier and Cost**	**Advantages**	**Disadvantages**
Luxury Type 60 mL Nalgene® Bottles (16) (One Piece Tip / Cap)	**Numerous Suppliers (17) ≈ $ 70 per dozen**	**High Quality; Easy to Refill; Drops Easy to Control (Hard Squeeze)**	**Large Drop Size (15 Drops per g) Cap Not Secure for Storage Hard to Squeeze, Tiring**
Economy Type 60 mL Wheaton® Bottles (18) (Snap-Out Inner Tip)	**Boreal-Northwest (19) ≈ $ 30 per dozen**	**Good Quality Small Drop Size (30 Drops per g) Cap Secure for Storage**	**Hard to Refill Drops Hard to Control (Light Squeeze)**
Supersaver Type Reusable Eye-Drop Bottles (10 mL and 15 mL)	**Reusable Eye-Drop Solution Bottles (Prescription or Over-the-Counter)**	**Good Quality Small Drop Size (30 Drops per g) Cap Secure for Storage**	**Hard to Refill Drops Hard to Control Must be Refilled More Often**

Photograph: Polymer Drop-Dispensing Squeeze-Bottles (Credit D. Cash 2012)		
Left: Two 60 mL Nalgene Bottles: One-Piece Integrated Tip and Cap	**Centre: Three 60 mL Wheaton Bottles: Inner Snap-Out Tip and Outer Closed Cap**	**Right: 15 mL and 10 mL Eye-Drop Bottles: Inner Snap-Out Tips Shown**

6. Calculations

For this kind of analysis of a fruit juice, it is conventional to do calculations as if only the predominant acid were present, and to state the result in **g / L** units of that acid. For example, in the analysis of apple juice performed by Mike Jansen's students (end of **Section 3** above), the result should be calculated and reported as if only **malic acid** were present. The relevant experimental measurement is the **2.67 g** mean titration value obtained for **5 mL** samples of Tim Hortons apple juice.

Calculation of the amount of acid present in a juice or a drink must begin with a balanced chemical equation of the reaction. The relevant equation for the titration of the dicarboxylic acid malic acid is:

$$\textbf{(Malic Acid) } C_2H_3(OH)(COOH)_2 + 2\ NaOH \rightarrow Na_2C_2H_3(OH)(COO)_2 + 2\ H_20$$

Units for Calculations
Suitable units of concentration for the titrating solution in a gravimetric titration are **mol / kg** or more commonly **mmol / g**. For very high precision analyses all calculations would use this unit. Note that the solution concentration does not change with temperature, an advantage of gravimetric titrations.

However, as shown in **Table 5** the density of a 0.1 M NaOH solution is within a few percent of **1 g / mL**. For student titration calculations with 0.1 M NaOH solution it is a reasonable approximation to take the concentration as either **mmol / mL** or **mmol / g** as convenient.

Table 5: Density of ≈ 0.1 M NaOH Solution (20 ºC)	
CRC Handbook (20): 0.5 % NaOH solution	1.0037 g / mL
Boreal–Northwest MSDS (11): 0.1 M (0.4 %) NaOH solution	1.002 g / mL

Thus: **0.1 mol / L = 0.1 mmol / mL ≈ 0.1 mmol / g**

Sample Calculations
First, calculate the mass of malic acid (symbol **MA** below) in mg units that is equivalent to 1 g of the 0.1 M NaOH solution (**Note 5**). The molar mass of malic acid is **134.1 g / mol** (**134.1 mg / mmo**l). Unless the sodium hydroxide solution has been standardized, use **0.1 mmol / g** as an exact value. Use the balanced equation above:

$$\mathbf{1\ g\ 0.1\ M\ NaOH\ =\ 1\ g \times \frac{0.1\ mmol\ NaOH}{g} \times \frac{1\ mmol\ MA}{2\ mmol\ NaOH} \times \frac{134.1\ mg\ MA}{mmol\ MA}\ =\ 6.71\ mg\ MA}$$

Next, calculate the equivalent **g / L** concentration of the malic acid in a **5 mL** sample of apple juice. The units of this value are **g / L malic acid per 1 g of titrant**:

$$\mathbf{{}^{g}/_{L}\ MA\ per\ 1\ g\ titrant\ =\ \frac{6.71\ mg\ MA}{1\ g\ 0.1\ M\ NaOH} \times \frac{1}{5\ mL} \times \frac{1000\ mL\ Apple\ Juice}{L\ Apple\ Juice} \times \frac{1\ g\ MA}{1000\ mg\ MA}\ =\ 1.34\ {}^{g}/_{L}\ MA\ per\ g\ titrant}$$

Lastly, use the mean titration value of **<u>2.67 g</u>** of titrant mass for **<u>5 mL</u>** samples to calculate the **g / L** value of malic acid concentration in the apple juice:

$$\mathbf{{}^{g}/_{L}\ MA\ =\ 1.34\ {}^{g}/_{L}\ MA\ per\ g\ titrant \times 2.67\ g\ titrant\ =\ 3.58\ {}^{g}/_{L}\ MA}$$

Note 5: Choice of the **Calculation Method**
Of all possible calculation methods, this route has been chosen because it leads naturally to the value of the slope of the plot shown. The result of the second calculation above is the slope of the plot shown below.

As an alternative to calculations, the students could be given or could construct and use a linear plot as shown to determine the acid concentration in the drink from the mean titration mass. This might be most appropriate if you are analysing only one sample type. Some data for the other acids in juices and drinks and calculated values are given in **Table 6** below; these values may be helpful for student use.

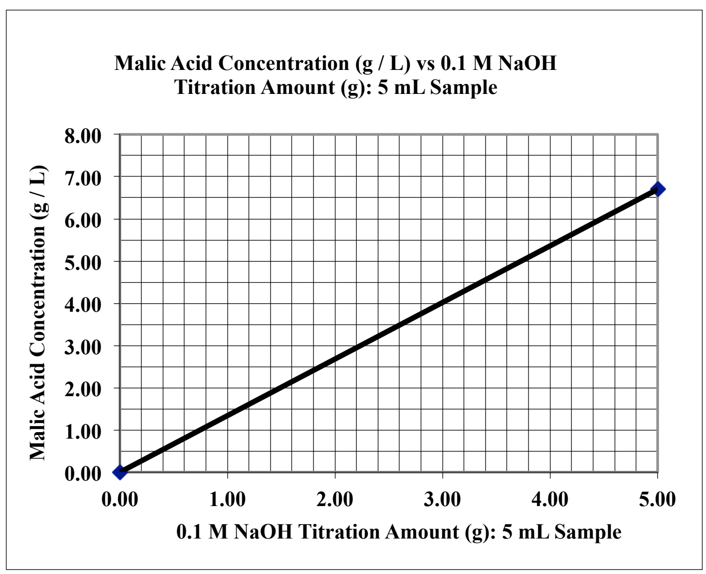


Table 6: Carboxylic Acid Data and Calculated Values per 1 g of 0.1 M NaOH Titrant Solution					
Acid	**Acid Type**	**mmol Mass**	**mg Equivalent**	**10 mL Samples**	**5 mL Samples**
Citric Acid	**tricarboxylic**	**192.1 mg**	**6.40 mg**	**0.640 g / L**	**1.28 g / L**
Tartaric Acid	**dicarboxylic**	**150.1 mg**	**7.51 mg**	**0.751 g / L**	**1.50 g / L**
Malic Acid	**dicarboxylic**	**134.1 mg**	**6.71 mg**	**0.671 g / L**	**1.34 g / L**
Succinic Acid	**dicarboxylic**	**118.1 mg**	**5.91 mg**	**0.591 g / L**	**1.18 g / L**
Phosphoric Acid	**triprotic**	**98.00 mg**	**3.27 mg**	**0.327 g / L**	**0.654 g / L**

7. Acknowledgements

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