

*Higher School Certificate Chemistry*

**PRACTICAL REPORT**

*Volumetric Analysis*

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## Volumetric Analysis

### Aim

To determine the concentration and percentage composition (weight per volume) of ethanoic acid in a 10-millilitre sample of vinegar, using volumetric analysis methods.

### Method

#### **Equipment**

##### Glassware

- Volumetric flask
- Burette
- Pipette
- Conical flask
- Measuring cylinder
- Multiple beakers to transfer liquids

##### Chemicals

- Monosodium dihydrogen phosphate ( $\text{NaH}_2\text{PO}_4$ )
- Sodium hydroxide (  $\text{NaOH}$  ) solution
- Vinegar sample

##### Miscellaneous

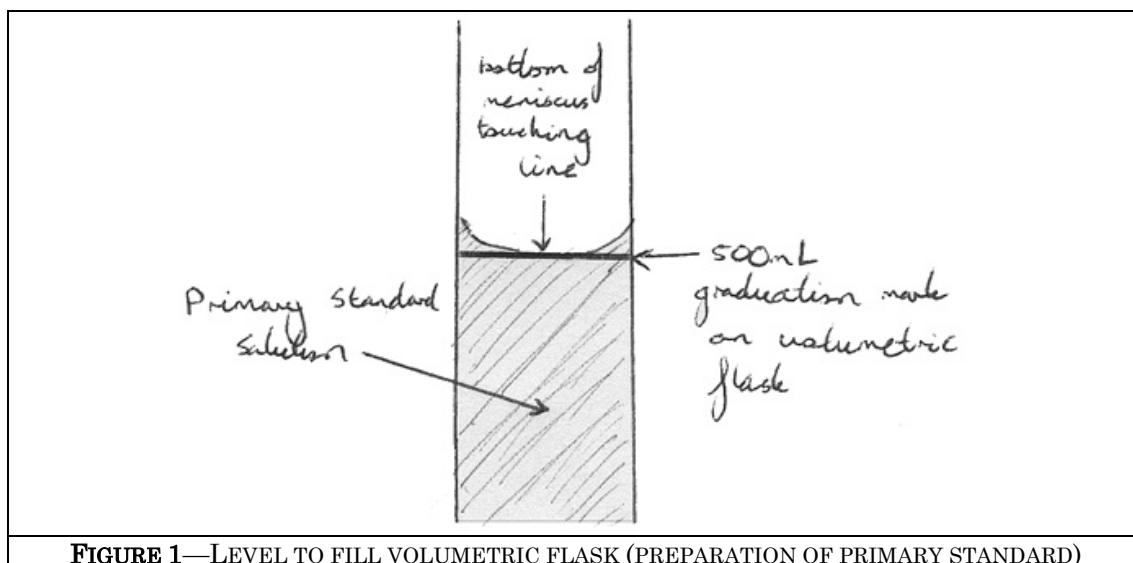
- Scales
- Watch glass
- Distilled water
- Funnel
- Wash bottle
- Pipette bulb
- Burette clamp
- Retort stand
- Indicator—phenolphthalein

#### **Procedure**

##### Making a Primary Standard—500mL of $0.04 \text{ mol.L}^{-1} \text{ NaH}_2\text{PO}_4$

1. Rinse volumetric flask thoroughly with distilled water several times.
2. Collect 2.40g of monosodium dihydrogen phosphate on watch glass (this is the mass required to achieve desired molarity—calculations used to arrive at this figure are shown under *Results*, p.5).
3. Use a funnel to place monosodium dihydrogen phosphate powder in volumetric flask.

4. Use wash bottle filled with distilled water to wash any of the monosodium dihydrogen phosphate powder from the funnel into the volumetric flask and add sufficient distilled water to dissolve all powder within flask.
5. Add stopper to flask and swirl liquid in flask until powder is completely dissolve.
6. Fill the volumetric flask with distilled water so that the bottom of the meniscus lies at the graduation mark of the flask. Distilled water should be dispensed by a wash bottle as the graduation mark is approached. See Figure 1, below.

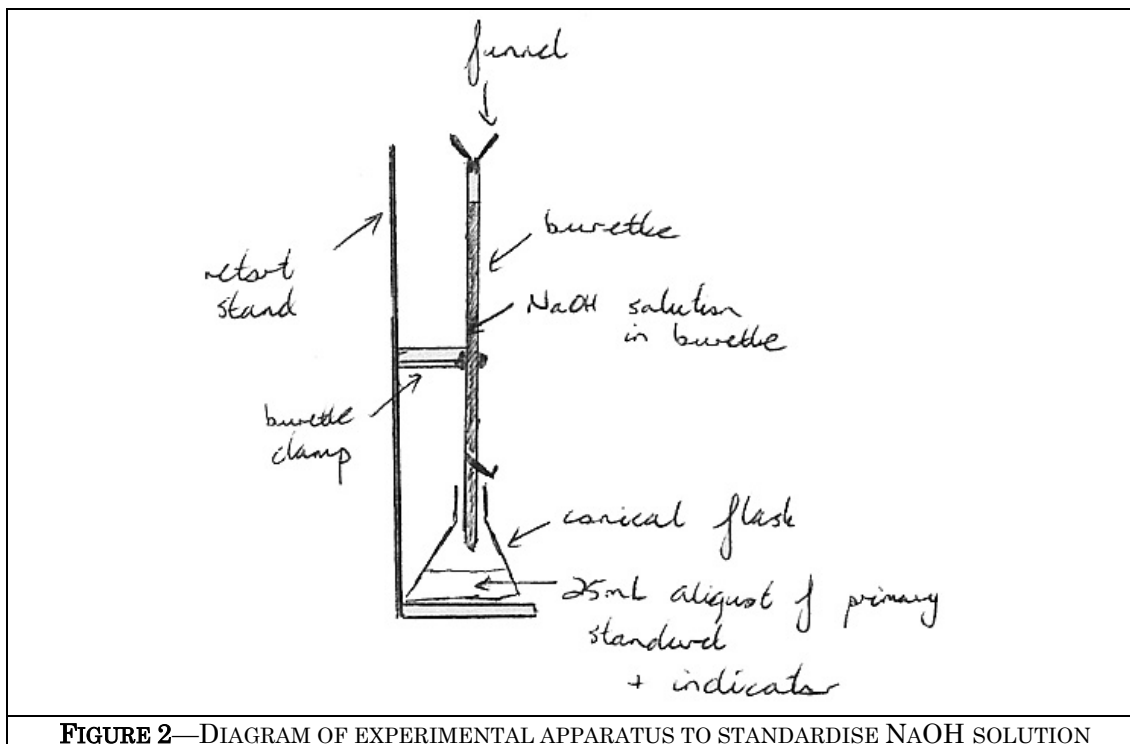


**FIGURE 1**—LEVEL TO FILL VOLUMETRIC FLASK (PREPARATION OF PRIMARY STANDARD)

7. Add stopper again and invert flask at least 10 times.

Standardising a Sodium Hydroxide solution

1. Rinse burette and pipette with distilled water.
2. Rinse burette with small amount of NaOH solution to be used
3. Rinse pipette with small amount of Primary Standard
4. Set up the experimental apparatus as shown in Figure 2, below.

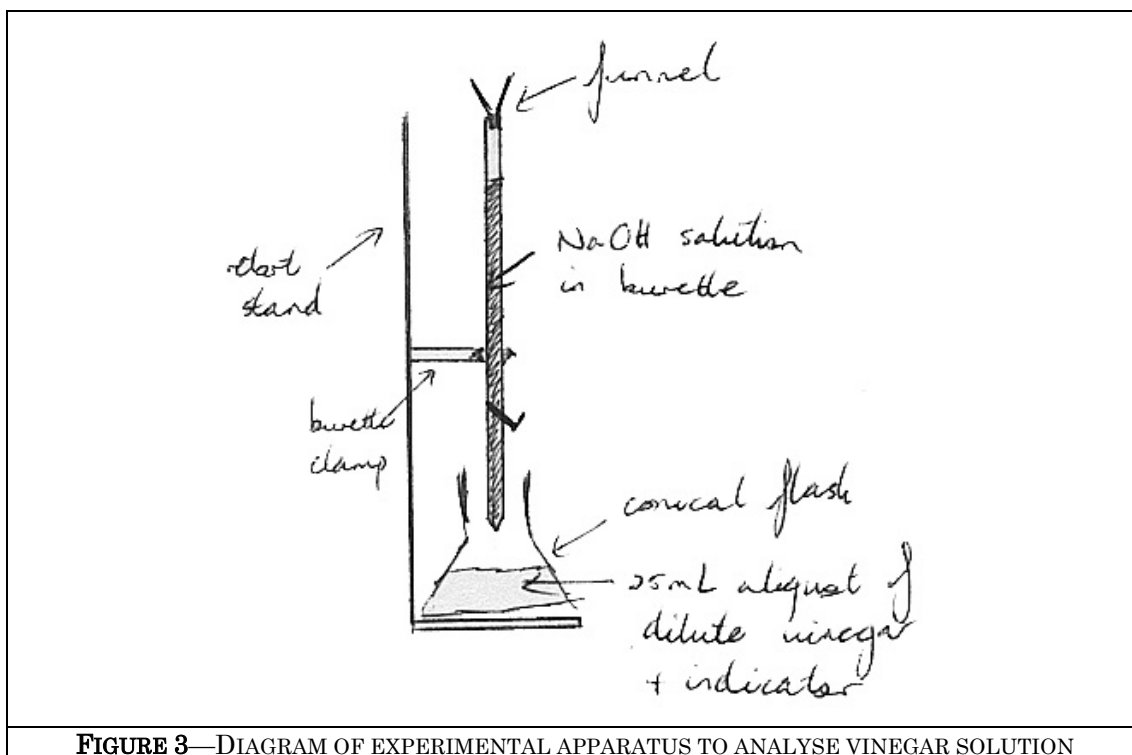


**FIGURE 2**—DIAGRAM OF EXPERIMENTAL APPARATUS TO STANDARDISE NaOH SOLUTION

5. Ensuring that there is solution below the level of the burette's tap, fill the burette with 50mL of NaOH.
6. Rinse conical flask with distilled water.
7. Use pipette to transfer one 25mL aliquot of Primary Standard to conical flask.
8. Add 3 drops of phenolphthalein indicator to conical flask.
9. Let NaOH solution drain into conical flask until there is a permanent colour change in the phenolphthalein indicator from clear to pink. Record this volume (this measure is recorded as the "rough titration").
10. Repeat steps 5—9 at least two more times, ensuring that as the volume attained by the "rough titration" is approached, the NaOH solution is drained more slowly, so that the end point of the reaction is not overshoot. Record results as titrations 1, 2, 3, etc.
11. Calculate, as shown in *Results*, pp.5–6, the concentration of the NaOH solution.

Analysing the vinegar

1. Into a measuring cylinder, place 10mL of vinegar. Then, dilute this to a volume of 100mL with distilled water.
2. Rinse pipette with distilled water.
3. Rinse pipette with small amount of diluted vinegar solution.
4. Ensure equipment is set up as shown in Figure 3, below.



**FIGURE 3**—DIAGRAM OF EXPERIMENTAL APPARATUS TO ANALYSE VINEGAR SOLUTION

5. Ensuring that there is solution below the level of the burette's tap, fill the burette with 50mL of standardised NaOH.
6. Rinse conical flask with distilled water.
7. Use pipette to transfer one 25mL aliquot of diluted vinegar solution to conical flask.
8. Add 3 drops of phenolphthalein indicator to conical flask.
9. Let NaOH solution drain into conical flask until there is a permanent colour change in the phenolphthalein indicator from clear to pink. Record this volume (this measure is recorded as the "rough titration").
10. Repeat steps 5—9 two more times, ensuring that as the volume attained by the "rough titration" is approached, the NaOH solution is drained more slowly, so that the end point of the reaction is not passed. Record results as titrations 1 and 2.
11. Calculate, as shown, in *Results* p.7, the concentration and percentage composition (w/v) of ethanoic acid in the original vinegar solution.

## Results

### Preparation of primary standard

#### Calculations:

$$c = 0.04, V = 0.5, n = ?$$

$$c = \frac{n}{V}$$

$$n = cV$$

$$\begin{aligned} n_{\text{NaH}_2\text{PO}_4} &= 0.04 \times 0.5 \\ &= 0.02 \text{ mol} \end{aligned}$$

$$n = \frac{m}{M}$$

$$m = nM$$

$$= 0.02 \times 119.98$$

$$= 2.3996 \text{ g}$$

$$\approx 2.40 \text{ g}$$

That is, 2.40g of monosodium dihydrogen phosphate is required to produce 500mL of the 0.04 mol.L<sup>-1</sup> acidic solution.

### Standardising NaOH solution

The volumes of NaOH needed to cause a permanent colour change in the phenolphthalein indicator are shown in Table 1, below.

	<i>Volume of NaOH used (mL)</i>
Rough titration	2.2
1 <sup>st</sup> titration	2.2
2 <sup>nd</sup> titration	2.1
3 <sup>rd</sup> titration	2.1
	$\bar{X} = 2.13$

**TABLE 1**—RESULTS OF STANDARDISING NAOH SOLUTION

#### Calculations:

With regards to the aliquot of primary standard (NaH<sub>2</sub>PO<sub>4</sub>):

$$c = 0.04, V = \frac{25}{1000}, n = ?$$

$$c = \frac{n}{V}$$

$$n = cV$$

$$\begin{aligned} \therefore n_{\text{NaH}_2\text{PO}_4} &= 0.04 \times \frac{25}{1000} \\ &= 1 \times 10^{-3} \text{ mol} \end{aligned}$$

Effective mole ratio of  $\text{NaH}_2\text{PO}_4$  reacting with the  $\text{NaOH}$  is 1:1 (see *Discussion* for explanation).

$$\therefore \text{ number of moles NaOH} = 1 \times 10^{-3} \text{ mol}$$

$$\text{Now: } n = 1 \times 10^{-3}, V = \frac{2.13}{1000}, c = ?$$

$$c = \frac{n}{V}$$

$$= \frac{1 \times 10^{-3}}{\frac{2.13}{1000}}$$

$$= 0.4695 \text{ mol.L}^{-1}$$

That is, the precise concentration of the sodium hydroxide solution is  $0.4695 \text{ mol.L}^{-1}$ .

### Analysis of vinegar

Volumes of NaOH needed to cause a permanent colour change in the solution containing the dilute vinegar and the phenolphthalein indicator are shown in Table 2, below.

	<i>Volume of NaOH used (mL)</i>
Rough titration	4.3
1 <sup>st</sup> titration	3.8
2 <sup>nd</sup> titration	3.8
	$\bar{X} = 3.8$

**TABLE 2**—RESULTS OF VINEGAR ANALYSIS

#### Calculations:

3.8 mL NaOH needed

$$n = cV$$

$$\begin{aligned} \therefore n_{\text{NaOH}} &= 0.4695 \times \frac{3.8}{1000} \\ &= 1.7841 \times 10^{-3} \text{ mol} \end{aligned}$$

Since mole ratio in reaction is 1:1;

$$\begin{aligned} n_{\text{ethanoic acid}} &= n_{\text{NaOH}} \\ &= 1.7841 \times 10^{-3} \text{ mol} \end{aligned}$$

$$V_{\text{ethanoic acid}} = 25 \text{ mL} = 0.025 \text{ L of dilute solution}$$

$$\begin{aligned} c_{\text{dilute}} &= \frac{1.7841 \times 10^{-3}}{0.025} \\ &= 0.071364 \end{aligned}$$

$$n_{\text{acid in concentrate}} = n_{\text{acid in dilute}}$$

$$\frac{c_{\text{vinegar}} \times 10}{1000} = \frac{0.071364 \times 100}{1000}$$

$$c_{\text{vinegar}} = 0.71364 \text{ mol.L}^{-1}$$

$$\text{Molar Mass}_{\text{ethanoic acid}} = 60 \text{ g.mol}^{-1}$$

$$\begin{aligned} \therefore 0.71364 \text{ mol}/100 \text{ mL} &= \frac{0.71364}{10} \times 60 \\ &= 4.28 \text{ g}/100 \text{ mL} \end{aligned}$$

$\therefore$  Percentage of ethanoic acid in vinegar sample was 4.28% (w/v)

## Conclusion

Through volumetric analysis methods, the concentration of ethanoic acid in the sample of vinegar was found to be  $0.71364\text{mol.L}^{-1}$ . The percentage composition of ethanoic acid in the vinegar sample was found to be 4.28% (weight per volume).

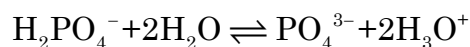
## Discussion

On the whole, there are a number of areas in which this practical could have been improved. Primarily, these areas for improvement stemmed from a lack of practice in volumetric analysis techniques and a poor understanding of the concepts involved. This was perfectly demonstrated when, instead of filling the burette with sodium hydroxide, I filled it with the Primary Standard. As the Primary Standard was only a weak acid, and the NaOH a strong base, the volume of Primary Standard required to neutralise the NaOH would have been huge, as was realised when the 50mL burette had emptied and the indicator had not yet changed colour. This, however, was easily remedied, and the reversal of these substances made the analysis possible. Indeed, just making this simple mistake once alerted me to the wide range of factors one must consider when undertaking volumetric analysis, and I believe I will not make mistakes of the same nature in the future.

When standardising the sodium hydroxide solution, the question arose as to the mole ratio of the primary standard's reaction with the sodium hydroxide. Within the primary standard, the reactions between the monosodium dihydrogen phosphate and water to form hydrogen ions are shown below:



If this acid were to be treated regularly, as a strong acid, it would be expected that the net reaction would be:



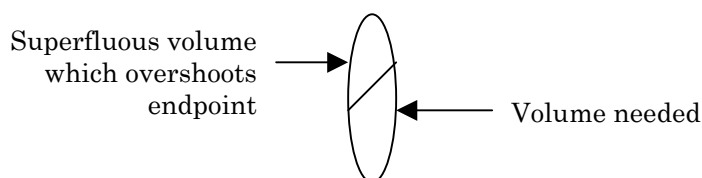
This would yield two hydrogen ions, meaning that the mole ratio of the primary standard reacting with the sodium hydroxide would have to be 2:1. After class discussion, however, it was realised that this could not be the case. One of the main factors which pointed towards the erroneousness of the 2:1 combining ratio was the fact that the sodium hydroxide solution was diluted to be approximately  $0.5\text{mol.L}^{-1}$ , and a 2:1 combining ratio would have yielded a concentration closer to  $1\text{mol.L}^{-1}$ .

Monosodium dihydrogen phosphate, however, is only a weak acid, and review of the acid dissociation constants ( $K_a$ ) for each of the reactions  $\textcircled{1}$  and  $\textcircled{2}$  (above) revealed why the ratio was not 2:1, instead, it is effectively 1:1. These values, which show the ratio of the concentrations of the products compared to the reactants, revealed that  $K_{a1}$  was equal to

$6.2 \times 10^{-8}$  and  $K_{a2}$  was equal to  $2.2 \times 10^{-13}$ . Such a large discrepancy between the two reactions means that the much lower  $K_a$  of ② makes the concentration of hydrogen ions that reaction produces so low that it can be omitted, meaning that only the hydrogen ion from ① is of any consequence. This would thus yield the correct combining ratio of 1:1.

Though the process of titration can be extremely accurate, there were a number of factors within this practical which could have served as sources of error in this volumetric analysis. Although each was relatively minor, the cumulative effect of all of them has the potential to produce markedly flawed results. The first of these sources for error was the way in which the primary standard was prepared. When the monosodium dihydrogen phosphate powder was placed into the volumetric flask, there was a small amount of powder residue which remained on the watch glass, which could have very slightly skewed the desired concentration of the Primary Standard acid, thus having a flow-on effect to the concentration of the standardised NaOH solution and the vinegar.

Another main source, not so much of error, but of inaccuracy, is the method by which the burette dropped the sodium hydroxide into the conical flask. As the indicator changes at a very specific pH, where the acidic and basic substances have neutralised each other, the increments by which the burette dripped the sodium hydroxide may have overshoot the exact endpoint. This concept can be most easily understood by looking at the following diagram, which shows a drop of the sodium hydroxide solution. Only the bottom half of the drop is required to achieve the endpoint, but it is obvious that despite this, the volume of the whole drop must leave the burette.



Yet another factor which *could* have been a source of error was the choice of indicator. It is important to choose an indicator that will undergo a sharp colour change at the exact endpoint of the titration. The pH change is slightly different for different strengths of acid and base so different indicators should be used. As the titrations involved in this experiment both involved a weak acid and a strong base, the indicator phenolphthalein was used, which changed markedly from clear to pink at the endpoint of the reaction.

Evidently, the most obvious application of this type of analysis, is to do as was done in this practical and find the percentage composition of a certain acidic or basic compound in a substance. Such techniques are widely used to determine impurities in food, or to make sure that the percentage composition of acid or base in acidic or basic foodstuffs remains within strict legal limits to ensure its suitability for consumption. One example of

such legal regulations is the approximate upper limit for the concentration of ethanoic acid in vinegar to be 4%. Volumetric analysis, however, can also be applied to environmental monitoring to ensure that the concentrations of potentially hazardous acidic and basic compounds in natural environments remain within acceptable parameters.

Taking into consideration the legal limit on the concentration of ethanoic acid in vinegars, the figure of 4.28% arrived at in this practical (within the approximate range of the 4% limit), reveals that the experimental techniques of volumetric analysis can and have been used with significant accuracy, to calculate the concentration and percentage composition of acidic or basic compounds within a substance.

## Bibliography

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