

# Experiment 10

## *Acetic Acid Content of Vinegar: An Acid-Base Titration*



## The task

The goal of this experiment is to determine accurately the concentration of acetic acid in vinegar *via* volumetric analysis, making use of the reaction of acetic acid with a strong base, sodium hydroxide.

## Skills

At the end of the laboratory session you should be able to:

- use an analytical balance,
- use a pipette filler and a pipette,
- use a volumetric flask to make up a solution of a given concentration accurately,
- use a burette to carry out a titration.

## Other outcomes

You will develop an understanding of how an acid-base indicator can be used to establish the end-point of a titration.

## The Assessment

You will be assessed on your ability to use the pipette and pipette filler. See Skill 4.3.



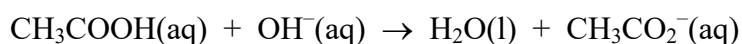
## Introduction

Vinegar is an acidic liquid, which is made both naturally and synthetically, from the oxidation of ethanol,  $\text{CH}_3\text{CH}_2\text{OH}$ , in an alcohol-containing liquid such as wine, fermented fruit juice (*e.g.* cider) or beer. It has been used since ancient times as an important cooking ingredient, *e.g.* in salad dressings and on fish and chips. The key chemical component of vinegar is acetic acid,  $\text{CH}_3\text{COOH}$  (systematic name: ethanoic acid). The trivial name, acetic acid, is derived directly from the word for vinegar, which, for example, in Italian is called *aceto*. The word vinegar itself derives originally from the Latin *vinum aegrum*, meaning “feeble wine”. In fact, when a wine has “gone off” and has acquired a sour taste, this is due to the oxidation of the ethanol in the wine to acetic acid. (The “corking” of wine, *i.e.* tainting of the wine by compounds transferred from or through the cork, is due to a totally different chemical process.) Vinegar derived from red or white wine is the most commonly used form of vinegar in Mediterranean countries and central Europe. The acetic acid content of vinegar can vary widely, but for table vinegar it typically ranges from 4 to 8 % v/v. When used for pickling, the acetic acid content can be as high as 12 %. The purpose of this experiment is to determine the acetic acid content of a commercial vinegar by volumetric analysis.

### *Volumetric analysis*

Volumetric analysis (see Skill 4) is a technique that employs the measurement of volumes to determine quantitatively the amount of a substance in solution. In any reaction between two or more species, the reaction equation shows the stoichiometric ratio of reacting species. Hence, if the concentration of one of the solutions is known, the concentrations of the others can be determined from the volumes used.

Take, for example, the reaction being investigated today, that between solutions of acetic acid and sodium hydroxide, NaOH. The reaction equation is:



The  $\text{OH}^-$  (from sodium hydroxide) is present at a known molar concentration. A fixed volume of  $\text{OH}^-$  solution is taken and the  $\text{CH}_3\text{COOH}$  solution progressively added until the point at which complete reaction of the substances is reached. This is called the equivalence point, *i.e.*, the point at which the moles of  $\text{CH}_3\text{COOH}$  added equals the number of moles of  $\text{OH}^-$  present in the original solution. The incremental process of adding the  $\text{CH}_3\text{COOH}$  solution is called titration. It enables the concentration of the  $\text{CH}_3\text{COOH}$  to be determined from the stoichiometric equation and the volume of the  $\text{CH}_3\text{COOH}$  solution needed to reach the equivalence point.

### *pH Indicators*

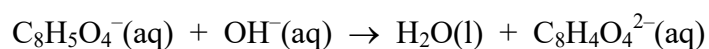
Up to the equivalence point the reacting solution contains an excess of one reactant (here  $\text{OH}^-$ ); after the equivalence point the solution contains an excess of the other reactant (here  $\text{CH}_3\text{COOH}$ ). We need some means of observing this switch-over. In acid/base titrations (to which the acetic acid/hydroxide titration belongs), the change in the colour of a pH indicator is often used to identify the switch-over. The point at which this observed change occurs is called the end point of the reaction. An appropriate indicator is one in which the end point and the equivalence point are as close together as possible.

pH indicators themselves are generally both weak acids and strongly coloured dyes, which respond with a dramatic change in colour when they react with base, *i.e.*  $\text{OH}^-$ . Because they react with base, in principle they compete with the acid whose concentration you are trying to measure (*i.e.*  $\text{CH}_3\text{COOH}$ ). However, because the indicators are so highly coloured, very low concentrations are required, and you can generally ignore perturbation of the titration by the indicator in your calculations. However, it is important to keep the indicator concentration low.

In this experiment you will use the indicator phenolphthalein, which undergoes a sudden change in colour from pink in basic solution to colourless in acidic solution. The midpoint of the colour change occurs at a pH of 9.5, *i.e.* already slightly basic. This is a perfect choice as an indicator for the titration of acetic acid with hydroxide ion, because the solution at the equivalence point of this titration is slightly basic.

### *Standardisation*

Solutions of sodium hydroxide react slowly with carbon dioxide gas present in the atmosphere. Consequently, the solution of sodium hydroxide which you will use in today's experiment needs to be standardised (*i.e.* its concentration accurately determined) before use. This is done by titrating with a solution of a stable acidic compound of high purity which can be very accurately prepared to a known concentration. This is known as the primary standard. In this experiment you will use potassium hydrogenphthalate,  $\text{KC}_8\text{H}_5\text{O}_4$ , for this purpose. The equation for its reaction with hydroxide ion is:



In this experiment, you will perform a series of acid/base titrations. In the course of these titrations you will become familiar with the technique of titration and the calculations associated with volumetric analysis. Read the Skills sections on weighing by difference (Skill 3.2) and on volumetric analysis (Skill 4.1 - 4.8) before your practical session. An example titration and the relevant calculations can be found in Appendix 10.1.

## Safety

### *Chemical Hazard Identification*

**1 M sodium hydroxide** - hazardous. Corrosive, irritant. Avoid eye or skin contact.

**potassium hydrogenphthalate** - non-hazardous. Low toxicity, irritant.

**phenolphthalein** - moderate toxicity, irritant. Avoid eye and skin contact.

**vinegar** - non-hazardous

### *Risk Assessment and Control*

Moderate risk.

Pipettes are easily broken, resulting in dangerous jagged glass edges, especially if the pipette filler is used incorrectly. Read Skill 4.2 & 4.3 for its correct use and practise the techniques described before proceeding.

### *Waste Disposal*

All of the solutions used today can be washed down the sink with water. Solid waste should be thrown in the bin.

## Experimental

*This experiment is to be carried out individually.*

***NOTE: Failure to follow the correct procedures as explained in Skill 4 will result in wildly inaccurate results. The notes below do NOT give a full description of the techniques.***

### **Part A Standardisation of 0.1 M sodium hydroxide with the primary standard potassium hydrogenphthalate**

(A1) In a clean, dry 100 mL beaker take about 40 mL of the approximately 1 M solution of NaOH. Dilute the solution by a factor of ten by pipetting 25.00 mL of it into a 250 mL volumetric flask and making the volume up to 250.0 mL with deionised water. Swirl/shake/invert for about 2 minutes to mix the solution thoroughly.

(A2) Transfer this solution to a 250 mL conical flask and label the solution as 0.1 M NaOH. Retain this as your stock of 0.1 M sodium hydroxide for standardisation.

(A3) Use an analytical balance in the balance room at the back of the lab to weigh out accurately about 4 g (anywhere in the range 3.9 – 4.1 g will do) of potassium hydrogenphthalate from the sample tubes provided into a clean, dry 100 mL beaker (see Skill 3.2). You need to know the mass accurately to  $\pm 0.0002$  g for your subsequent calculations. **Record the exact mass in your logbook.**

(A4) Back at your bench, add about 80 mL of deionised water to the potassium hydrogenphthalate solid and stir to dissolve it. Transfer the solution to a 250 mL volumetric flask with the aid of the stirring rod and a plastic funnel. Add some more deionised water to the flask until just below the mark, mix to homogenise the solution and then make the solution up to the mark with deionised water, adding the water with a dropper. (**Note:** Never add water directly from a wash bottle. You can easily go past the mark and you'd have to throw the solution away and start again). Mix. Transfer the solution to a 250 mL conical flask and label it as  $\text{KC}_8\text{H}_5\text{O}_4$ .

***For your logbook:***

*Calculate the molar concentration of the potassium hydrogenphthalate,  $\text{KC}_8\text{H}_5\text{O}_4$ , solution.*

(A5) Read Skill 4.8. Use a pipette to transfer 25.00 mL of your 0.1 M NaOH solution to a conical flask. Add 2 drops of phenolphthalein indicator and swirl to mix. Carry out a rapid titration with your  $\text{KC}_8\text{H}_5\text{O}_4$  solution in the burette, gently swirling the conical flask as you titrate. **In your logbook record the rough volume required to reach the endpoint** (*i.e.* the volume required to just observe a persistent colour change).

(A6) Pipette another 25.00 mL of your 0.1 M NaOH into a clean conical flask. Add the indicator. Fill the burette again with the  $\text{KC}_8\text{H}_5\text{O}_4$  solution and **record the initial volume in the burette (in mL to two decimal places) in your logbook**. Then titrate the  $\text{KC}_8\text{H}_5\text{O}_4$  solution from the burette into the NaOH solution, slowing down to drop by drop and then split drops about 2 mL before the rough titration volume from your last titration. **Record the final volume of  $\text{KC}_8\text{H}_5\text{O}_4$  in the burette at the endpoint and hence calculate the total volume of  $\text{KC}_8\text{H}_5\text{O}_4$  solution added (the titre value) in your logbook.**

(A7) Repeat accurate titrations (as described in (A6)) until you have 3 concordant titre values that are within 0.10 mL of one another.

***For your logbook:***

*Calculate the exact concentration of your roughly 0.1 M solution of NaOH. Note: A sample calculation is given in Appendix 10.1.*

*Check your concentration with your demonstrator before proceeding to Part B.*

## **Part B Titration of vinegar with standard sodium hydroxide**

(B1) Dilute one of the vinegars by a factor of ten by pipetting 25.00 mL of it into a 250.0 mL volumetric flask and making it up to the mark with deionised water. Label the solution, *e.g.* dilute vinegar or dil.  $\text{CH}_3\text{COOH}$  (just as long as you know what it is). **Record in your logbook which particular type of vinegar you used.**

(B2) Perform a series of titrations with 25.00 mL of your standardised 0.1 M NaOH solution in the conical flask and the diluted vinegar in the burette (thoroughly rinsed from the last series of titrations). Don't forget to add 2 drops of indicator to your NaOH solution. As in Part A, the solution should start pink and your endpoint will be when the solution in the conical flask is persistently colourless on swirling. Repeat your titrations until you have 3 concordant titre values within 0.10 mL of one another. **Record your titre values in your logbook.**

***For your logbook:***

*Calculate the molar concentration of acetic acid in the vinegar you chose. Don't forget that you diluted the vinegar by a factor of 10 (i.e. you have to correct for this in your final calculation). (See Appendix 10.1 for a sample calculation.)*

*Calculate the concentration of acetic acid in your vinegar as a volume percentage. The density of pure acetic acid is  $1.049 \text{ g mL}^{-1}$  and its molar mass is  $60.05 \text{ g mol}^{-1}$ .*

*Does it fall within the standard range of 4-8 % v/v for table vinegars?*

**Group Discussion**

Collect on a whiteboard everyone's measurements of the volume percentages of acetic acid in the various vinegars.

Calculate the mean value of the volume percentage of acetic acid for each type of vinegar. Write these values in your logbook.

Which vinegar has the highest mean acetic acid content?

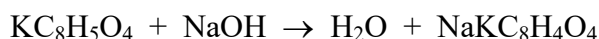
## Appendix 10.1: Sample Calculation

Primary standard consisted of 4.0982 g of  $\text{KC}_8\text{H}_5\text{O}_4$  dissolved in 250.0 mL water.

31.10 mL of this primary standard reacted with 25.00 mL of the NaOH solution.

22.45 mL of the diluted vinegar reacted with 25.00 mL of the NaOH solution.

The molecular equation for the reaction between potassium hydrogenphthalate and sodium hydroxide is:



Molar mass of  $\text{KC}_8\text{H}_5\text{O}_4$  is  $39.10 + (8 \times 12.01) + (5 \times 1.008) + (4 \times 16.00) = 204.22 \text{ g mol}^{-1}$

Mass of  $\text{KC}_8\text{H}_5\text{O}_4$  weighed out = 4.0982 g

Concentration of standard  $\text{KC}_8\text{H}_5\text{O}_4$  solution =  $\frac{4.0982 \text{ g}}{204.22 \text{ g mol}^{-1}} \times \frac{1}{0.250 \text{ L}} = 0.08027 \text{ M}$

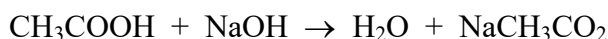
The mean titre value for the NaOH/ $\text{KC}_8\text{H}_5\text{O}_4$  titration was 31.10 mL.

31.10 mL of  $\text{KC}_8\text{H}_5\text{O}_4$  sol'n contains  $0.08027 \text{ mol L}^{-1} \times 0.03110 \text{ L} = 0.002496 \text{ mol KC}_8\text{H}_5\text{O}_4$

Hence 25.00 mL of NaOH solution contains 0.002496 mol of NaOH

Hence concentration of NaOH solution =  $\frac{0.002496 \text{ mol}}{0.0250 \text{ L}} = 0.09986 \text{ M}$

The molecular equation for the reaction between acetic acid and sodium hydroxide is:



The mean titre value for the NaOH/ $\text{CH}_3\text{COOH}$  titration was 22.45 mL.

25.00 mL of NaOH solution contains  $0.09986 \text{ mol L}^{-1} \times 0.0250 \text{ L} = 0.002496 \text{ mol NaOH}$ .

Hence 22.45 mL of diluted vinegar contains 0.002496 mol of  $\text{CH}_3\text{COOH}$

Hence concentration of diluted vinegar =  $\frac{0.002496 \text{ mol}}{0.02245 \text{ L}} = 0.1112 \text{ M}$

Hence concentration of undiluted vinegar =  $10 \times 0.1112 \text{ M} = 1.112 \text{ M}$

Therefore 1.000 L of undiluted vinegar contains 1.112 mol of  $\text{CH}_3\text{COOH}$ .

1.112 mol of  $\text{CH}_3\text{COOH}$  has volume  $\frac{1.112 \text{ mol} \times 60.05 \text{ g mol}^{-1}}{1.049 \text{ g mL}^{-1}} = 63.66 \text{ mL}$

Volume percent (%v/v) is defined as  $\%v/v = \frac{V_{\text{solute}}}{V_{\text{solution}}} \times 100$

Hence %v/v concentration of vinegar is  $\frac{63.66 \text{ mL}}{1000 \text{ mL}} \times 100 = 6.366 \text{ %v/v}$