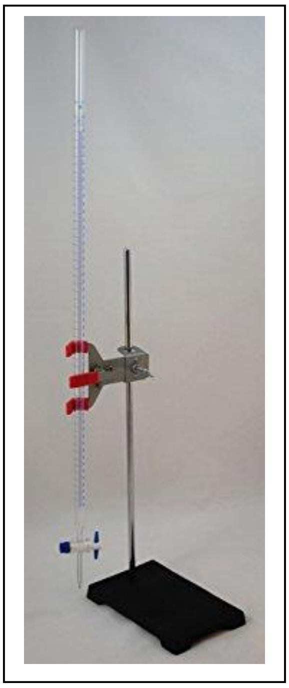
Lesson 1 Volumetric analysis

Click to revise volumetric analysis

There are many instances where we need to find the amount of acid or base present in a given volume of liquid. Volumetric analysis is a technique of finding unknown concentrations. This involves mixing two solutions, one with a known concentration, known as a ***standard solution***, and the other with an unknown concentration, which is usually the sample being analysed. The solutions are mixed using a burette until they have just reacted completely. This procedure is known as a titration. The point at which the solutions are mixed in just the right ratio to achieve complete reaction is called the ***equivalence point***. At this point the solutions of acid and base have been neutralised. Since the solutions used are often clear, indicators are used to identify the time during the reaction when neutralisation is achieved(***end point***). Indicators change colour at different pH levels and this alerts us to the fact that we are at the ***end point***.

Firstly some terminology
- Standard solution – solution whose concentration is known accurately
- Primary standard – is a substance used to prepare a standard solution

    A primary standard should:
    - be available in its pure form
    - have a known chemical formula
    - not react with the atmosphere
    - have a high molecular mass so as to minimise the percentage error when weighing.
- Equivalence point – is the point in the reaction when the reactants are mixed in exactly the mole ratio shown by the balanced chemical equation of the reaction.
- End point – the point during the titration at which the indicator undergoes a permanent colour change.
- Indicator – a chemical that changes colour at a pH close to the equivalence point of a titration
- Burette – glassware that delivers an accurate variable volume of solution.



- Titre – the volume delivered by the burette needed to reach the end point of the titration
- Concordant titres – titres that vary within 0.10mL from highest to lowest.
- Average titre – the average of the three concordant titres.

- Aliquot – the volume delivered by a pipette.

- Titration curve (pH curve)
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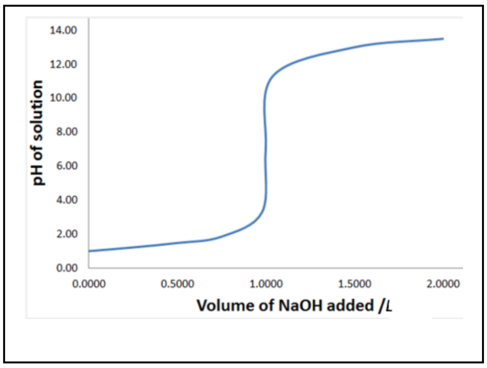
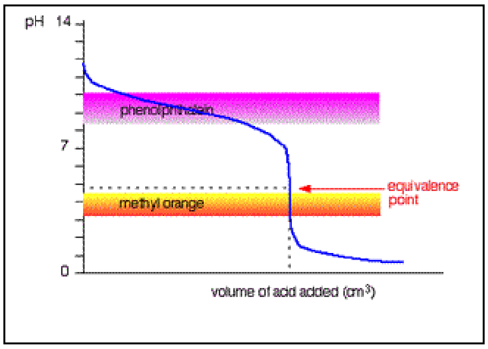


Indicators change colour within a very narrow pH range. It is important to use an indicator that changes colour very close to the equivalence point.

Consider the pH curve shown on the right. It shows the pH range of two indicators, methyl orange and phenolphthalein. Looking at the curve it is important to see that methyl orange should be used as the indicator for this titration as it will change colour close to the equivalence point.



When performing titrations it is important to note the impact of washing the glassware with water or different solutions. The following should be noted.
- the conical flask and volumetric flasks should be rinsed with deionised water.
- the burette and pipette should be rinsed with the solutions that they will deliver.

1) A student performed 5 titrations. The following titres were obtained.
   13.05, 12.95, 12.78, 13.12, 12.99.
   a) What is the average titre for this titration?
   (13.05 + 12.95 + 12.99) / 3 = 13.00 mL

   b) If a 0.110M Na_2CO_3 solution was used to fill the burette, how many mol of Na_2CO_3 was present in an average titre?
   n = C X V
   => 0.110 X 0.01300 = 1.43 X 10^{-3}mol

2) A student prepared a standard solution of sodium carbonate. A 250 mL volumetric flask was used to prepare a 0.110 M Na_2CO_3.
   a) What mass of Na_2CO_3 should be weighed and transferred into the volumetric flask?
   Step 1 find the number of mol of Na_2CO_3
   => n = C X V
   => 0.110 X 0.250 = 0.0275
   Step 2 find the mass of Na_2CO_3
   => 0.0275 X 106.0 = 2.92 g
   b) An unknown solution of vinegar was analysed for its acetic acid (CH_3COOH) concentration. 20.00 mL of the sample vinegar solution was pipetted into a 250 mL volumetric flask and made up to the mark using distilled water.
   The burette was filled with the standard solution prepared in part a) above. A 25.0mL aliquot of the diluted solution of vinegar was taken from the volumetric flask and placed in a conical flask with two drops of indicator.
   An average titre of 10.50 mL was obtained.
   The reaction taking place is given below.
   $Na_2CO_3(aq) + 2CH_3COOH(aq) \rightarrow 2NaOOCCH_3(aq) + H_2O(l) + CO_2(g)$
      i. Calculate the mol of Na_2CO_3 present in the average titre.
      0.110 X 0.01050 = 1.16 X 10^{-3} mol
      ii. Calculate the mol of CH_3COOH present in the conical flask
      1.16 X 10^{-3} X 2 = 2.32 X 10^{-3} mol

iii. Calculate the mol of CH_3COOH present in the volumetric flask

(250/25) X 2.32 X 10^{-3} mol = 2.32 X 10^{-2}mol

iv. Calculate the concentration of acetic acid, in mol/litre and in %v/v, in the original vinegar solution. (density of acetic acid = 1.05 g/mL)

Since 2.32 X 10^{-2}mol of acetic acid came from 20.0 mL of the sample of vinegar then the concentration is

=> C = n/V => 2.32 X 10^{-2} / 0.0200 = 1.16M

or

mass of acetic acid in 20.0mL of vinegar is

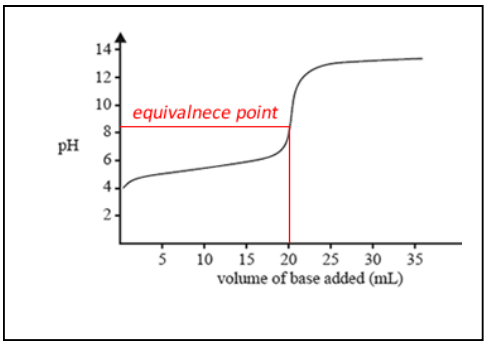
=> 2.32 X 10^{-2} X 60.05 = 1.39 g

Volume of acetic acid in 20.0 mL of vinegar is

=> 1.39g / 1.05g/mL = 1.32 mL

%v/v => (1.32 /20.0) X 100 = 6.60% v/v

3) The change in pH as a 0.100 M solution of NaOH is added to a 40.0 mL solution of acetic acid in a conical flask, is shown on the right.



a) Write a balanced chemical equation for the reaction between NaOH and CH_3COOH.

$CH_3COOH(aq) + NaOH(aq) \rightarrow NaOOCCH_3(aq) + H_2O(l)$

b) Using the data sheet select an appropriate indicator for this titration. Explain your selection.

Methyl Blue with a pH range of 8.0 – 9.6

c) Suggest why the equivalence point of a titration is not always at pH 7

It depends on the conjugate acid or base that is produced. In this case the conjugate base for the weak acetic acid is a relatively strong acetate ion (CH_3COO^-). Hence the solution at the equivalence point is basic.

d) What is the concentration of the acetic acid solution present in the conical flask?

Step 1 find the mol of NaOH added.

=> n = C X V = 0.100 X 0.020 = 0.0020 mol

Step 2 find the mol of acetic acid in 40.0 mL

=> 0.0020 mol

Step 3 find the concentration in mol/litre

=> 0.0020 / 0.0400 = 0.050 M

e) Consider the following.

i. Washing the conical flask with distilled water.

ii. Washing the burette with distilled water.

iii. Washing the pipette with distilled water.

iv. Washing the burette with NaOH solution

v. Washing the conical flask with NaOH solution.

Which of the above would give.

i. a titre less than expected. *iii, v.*

ii. will have no effect on the average titre obtained. *i, iv*

iii. will give a titre more than expected. *ii,*

4) State the difference between

i. a pipette and a burette

A burette delivers an accurate variable volume of solution.

A pipette delivers an accurate fixed volume of solution

ii. end point and equivalence point

The equivalence point is the point in the titration where reactants are mixed in the right stoichiometric ratio.

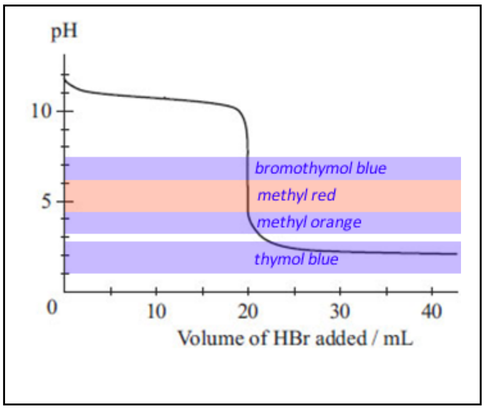
The end point is the point is the titration where the indicator undergoes a permanent colour change.

iii. titre and aliquot

A titre is the volume of solution delivered through a pipette until the indicator changes colour.

An aliquot is the volume of solution delivered via a pipette.

5) A 30 mL pipette was used to deliver an accurate volume of an unknown solution ("X") containing methylamine into a 250 mL volumetric flask. Deionised water was used to fill the volumetric flask to the mark.
A 20.00 mL aliquot of this solution was placed in a 100 mL conical flask with two drops of an appropriate indicator and titrated against a 0.100 M HBr solution. The change in pH during the titration is shown on the right.



a) Write a balanced chemical equation for the reaction between HBr and CH_3NH_2.

$CH_3NH_2(aq) + HBr(aq) \rightarrow CH_3NH_3^+(aq) + Br^-(aq)$

b) Using the data sheet, which indicator, of the ones shown below, should be used for this titration? Explain why

i. Bromothymol blue

ii. Methyl orange

iii. Thymol blue

iv. Methyl red

Bromothymol blue and to a lesser extent methyl red. These indicators change colour around the steepest part of the titration curve at 20 mL of HBr. The equivalence point and the end point are very close.

c) Calculate the concentration of the original methylamine solution if the average titre was 20.00 mL.

⇨ *0.833M*

d) How would the result in c) above change if the:

i. burette was washed with water. *Higher*

ii. conical flask was washed with water *no change*

iii. conical flask was washed with HBr solution *Lower*

6) A 5.30 gram sample of brick cleaner was placed in a 200 mL volumetric flask and made up to the mark with distilled water. A 20.00 mL aliquot of the resulting solution was taken from the volumetric flask and placed in a 100 mL conical flask with two drops of an appropriate indicator and titrated against a 0.0212 M Na_2CO_3. An average titre of 10.10 mL was needed to reach the end point.
Calculate the concentration of HCl, in %w/w, in the cleaner.

Step 1 – Calculate the mol of Na_2CO_3 in the average titre.

=> $n = 0.0212 \times 0.01010 = 2.14 \times 10^{-4}$ mol

Step 2 – calculate the mol of HCl present in the conical flask.

=> $Na_2CO_3(aq) + 2HCl(aq) \rightarrow 2NaCl(aq) + H_2O(l) + CO_2(g)$

=> 4.28×10^{-4} mol

Step 3 – calculate the mass of HCl in the conical flask

=> mass = $36.5 \times 4.28 \times 10^{-4} = 1.56 \times 10^{-2}$ g

Step 4 – calculate the mass of HCl in the volumetric flask

=> 1.56×10^{-2} g $\times$ (200/20) = 0.156 g

Step 5 – calculate the concentration of HCl in %w/w

=> *(0.156/5.30) X 100 = 2.94% w/w*