

Name:

Volumetric Analysis	Objectives
1. Concentrations of Solutions	<ul style="list-style-type: none"> -define solution -define concentration -define molarity -express concentration of solutions in mol/L(molarity), g/L and also in % (w/v), % (v/v), % (w/w) -appreciate the everyday use of % v/v e.g. in alcoholic beverages -calculate molarity from concentration in grams per litre and vice versa -calculate number of moles from molarity and volume -perform simple calculations involving percentage concentrations -calculate the effect of dilution on concentration -apply knowledge of concentrations of solutions to everyday examples -describe how colour intensity can be used as an indicator of concentration -Define a primary standard and a standard solution -prepare standard solution of sodium carbonate
3. Volumetric Analysis	<p><i>(balanced equations will be given in all volumetric problems)</i></p> <ul style="list-style-type: none"> -identify appropriate apparatus used in volumetric analysis -use correct titrimetric procedure when carrying out titrations -solve volumetric problems from first principles -carry out a titration between hydrochloric acid and sodium hydroxide solutions and use this titration to make a sample of sodium chloride (OL only) -standardise a hydrochloric acid solution using a standard solution of sodium carbonate -calculate the relative molecular mass of a compound and of the amount of water of crystallisation in a compound from titration data <i>(balanced equations will be given in all volumetric problems)</i> -determine the concentration of ethanoic acid in vinegar -determine the amount of water of crystallisation in hydrated sodium carbonate

Defⁿ: A **solution** is a homogeneous mixture of a solute and a solvent.

Defⁿ: The **concentration** of a solution is the amount of solute dissolved in a given volume of the solution.

1. **% (w/w)**

Means grams of solute per 100 g of solution, or g/100g.

E.g. saline solution, 1% (w/w) NaCl = 1g NaCl in 100cm³ solution.

2. **% (w/v)**

Means grams of solute per 100 cm³ of solution, or g/100 cm³.

E.g. 37% (w/v) HCl = 37g HCl in 100cm³ solution.

3. **% (v/v)**

Means volume of solute per 100 cm³ of solution, or cm³/100 cm³.

E.g. Alcoholic drinks – Vodka's alcohol (ethanol) concentration is 37.5% (v/v) = 37.5 cm³ ethanol in 100 cm³ vodka.

4. **Parts per Million (ppm)**

Means the concentration in mg/L. This is calculated by multiplying the concentration in g/L by 1000.

1000mg = 1g.

5. Molarity (M or mol/L)

This is the main unit used in the chemistry course. This means the number of moles of solute in 1 litre of solution. E.g. A 0.125 M solution of KMnO_4 has 0.125 moles of KMnO_4 in 1 litre of litre solution.

Calculations**(a) mol/L to g/L**

- How many grams of FeSO_4 are present in a solution marked 0.35 M FeSO_4 ?

$$M_r \text{ of } \text{FeSO}_4 = (56) + (32) + 4(16) = 152$$

We have 0.35 mol/L FeSO_4

$$\times 152 \quad [\text{mol to g} \rightarrow \times M_r]$$

$$= 53.2 \text{ g/L}$$

- Calculate the concentration in grams per litre of bench hydrochloric acid whose concentration is 12 mol/L.

$$M_r \text{ of HCl} = (1) + (35.5) = 36.5$$

We have 12 mol/L HCl

$$\times 36.5 \quad [\text{mol to g} \rightarrow \times M_r]$$

$$= 438 \text{ g/L HCl}$$

(b) g/L to mol/L

- What is the molarity of a solution that contains 7.36 g of NaOH per litre of solution?

$$M_r \text{ of NaOH} = (23) + (16) + (1) = 40$$

We have 7.36 g/L NaOH

$$\div 40 \quad [\text{g to mol} \rightarrow \div M_r]$$

$$0.184 \text{ mol/L NaOH}$$

- Calculate the concentration of a solution containing 45 g of sulphuric acid in a litre of solution.

$$M_r \text{ of H}_2\text{SO}_4 = 2(1) + (32) + 4(16) = 98$$

We have 45 g/L H_2SO_4

$$\div 98 \quad [\text{g to mol} \rightarrow \div M_r]$$

$$0.4592 \text{ mol/L H}_2\text{SO}_4$$

(c) How many moles in a certain volume, given the solution's molarity

- Calculate how many moles of CH_3COOH are present in 25 cm^3 of 0.55 M CH_3COOH .

We have 0.55 mol/L CH_3COOH

$$\frac{0.55}{1000} \times 25$$

$$= 0.01375 \text{ mol/} 25 \text{ cm}^3 \text{ CH}_3\text{COOH}$$

If question was phrased “Calculate how many **grams** of CH_3COOH are present in 25 cm^3 of 0.55 M CH_3COOH ”, we would then have to multiply 0.01375 by the M_r of CH_3COOH .

2. How many moles of HCl are present in 60 cm³ of 0.4 M HCl?

We have 0.4 mol/L HCl

$$\frac{0.4}{1000} \times 60$$

$$= 0.024 \text{ mol/60 cm}^3 \text{ HCl}$$

(d) Calculate moles per litre, given the mass of solute and volume of solution

1. Calculate the concentration in moles per litre of a solution containing 45 grams of sulphuric acid per 240 cm³ of solution.

$$M_r \text{ of H}_2\text{SO}_4 = 2(1) + (32) + 4(16) = 98$$

We have 45 g/240 cm³ H₂SO₄

$$\div 98 \quad [\text{g to mol} \rightarrow \div M_r]$$

$$= 0.4592 \text{ mol/240 cm}^3 \text{ H}_2\text{SO}_4$$

$$\frac{0.4592}{240} \times 1000$$

$$= 1.93 \text{ mol/L H}_2\text{SO}_4$$

2. 7.6 g of anhydrous Na₂CO₃ is dissolved in deionised water and made up to 300 cm³ of solution. Express the concentration of this solution in mol/L.

$$M_r \text{ of Na}_2\text{CO}_3 = 2(23) + (12) + 3(16) = 106$$

We have 7.6 g/300 cm³ Na₂CO₃

$$\div 106 \quad [\text{g to mol} \rightarrow \div M_r]$$

$$= 0.0717 \text{ mol/300 cm}^3 \text{ Na}_2\text{CO}_3$$

$$\frac{0.0717}{300} \times 1000$$

$$= 0.239 \text{ mol/L Na}_2\text{CO}_3$$

(e) Compound calculations (combinations of (a) to (d))

1. What mass of sodium hydroxide is contained in 25 cm³ of a 1.5 M solution of sodium hydroxide?

$$M_r \text{ of NaOH} = (23) + (16) + (1) = 40$$

We have 1.5 mol/L NaOH

$$\times 40 \quad [\text{mol to g} \rightarrow \times M_r]$$

$$= 60 \text{ g/L NaOH}$$

$$\frac{60}{1000} \times 25$$

$$= 1.5 \text{ g/25 cm}^3 \text{ NaOH}$$

2. What volume of 0.01 M KMnO₄ solution will contain 5 g of KMnO₄?

$$M_r \text{ of KMnO}_4 = (39) + (55) + 4(16) = 158$$

We have 0.01 mol/L KMnO₄

$$\times 158 \quad [\text{mol to g} \rightarrow \times M_r]$$

$$= 1.58 \text{ g/L KMnO}_4. \text{ This can be rewritten as } 1.58 \text{ g/1000 cm}^3 \text{ KMnO}_4$$

$$\div 1.58$$

$$= 1 \text{ g/632.9 cm}^3 \text{ KMnO}_4$$

$$\times 5$$

$$= 5 \text{ g/3164.6 cm}^3 \text{ KMnO}_4$$

Dilution of solutions

To find the volume of a concentrated solution needed to make a less concentrated solution, use the formula:

$$V_c \times M_c = V_d \times M_d$$

V_c = Volume of concentrated solution

V_d = Volume of dilute solution

M_c = Molarity of concentrated solution

M_d = Molarity of dilute solution

Examples:

1. What volume of 12 M HCl is needed to make up 500 cm³ of 3 M HCl solution?

$$\begin{aligned} V_c \times M_c &= V_d \times M_d \\ V_c \times 12 &= 500 \times 3 \\ \frac{500 \times 3}{12} &= V_d \\ 125 \text{ cm}^3 &= V_d \end{aligned}$$

2. 15 cm³ of 2 M HNO₃ solution is diluted to 250 cm³ in a volumetric flask. What is the new concentration of the nitric acid?

$$\begin{aligned} V_c \times M_c &= V_d \times M_d \\ 15 \times 2 &= 250 \times M_d \\ \frac{15 \times 2}{250} &= M_d \\ 0.12 \text{ M} &= M_d \end{aligned}$$

Standard Solutions

Defⁿ: A **standard solution** is one whose concentration is accurately known.

Defⁿ: A **primary standard** is a substance that can be directly weighed and used to make a standard solution. It must:

- Be available in a pure and stable solid state
- Be soluble in water
- Have a high molecular mass
- Be anhydrous (no water of crystallisation)

Primary Standards: Na₂CO₃, NaCl

Not Primary Standards: HCl – it is a gas

I₂ – it sublimes

H₂SO₄ – it absorbs moisture from air

NaOH – it absorbs CO₂ and moisture from air

KMnO₄ – it is reduced by sunlight

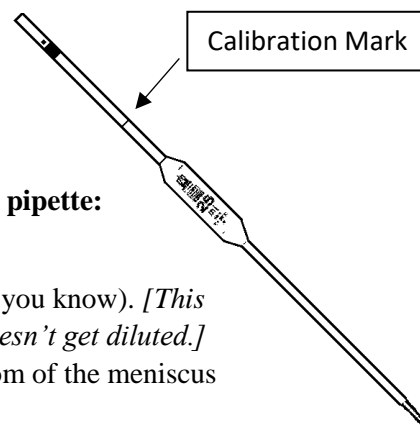
Equipment used in Titrations

1. Pipette:

Used to accurately measure a known volume of liquids/solutions.

Procedure for cleaning, filling and transferring solutions using a pipette:

- Rinse with deionised water.
- Rinse with the solution it is to contain (name this solution if you know). *[This is done to remove the water, so the solution in the pipette doesn't get diluted.]*
- Using a pipette filler, fill pipette with solution until the bottom of the meniscus reaches the graduation mark, at eye level.
- Let the pipette drain under gravity, touching the tip of the pipette against the flask to remove the last drop stuck to the tip.

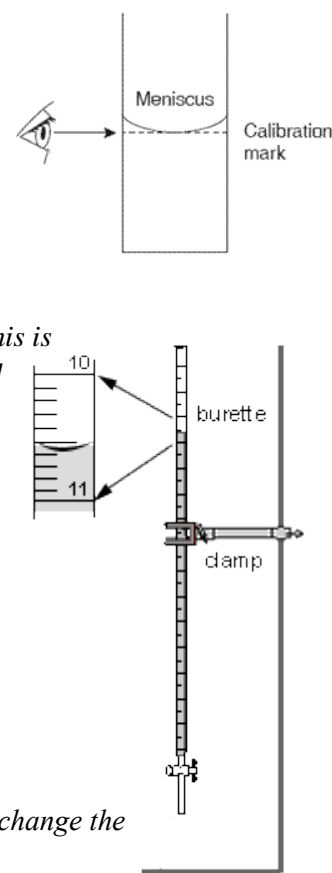


2. Burette:

Used to accurately measure the volume of liquid/solution added.

Procedure for cleaning and filling a burette:

- Rinse with deionised water.
- Rinse with the solution it is to contain (name this solution if you know). *[This is done to remove the water, so the solution in the burette doesn't get diluted.]*
- Clamp vertically.
- Using a funnel, fill the burette above the zero mark.
- Remove the funnel.
- Open the tap to bring the bottom of the meniscus to the zero mark, and to fill the jet below the tap.



3. Conical Flask:

A specially shaped flask that allows swirling without spilling the contents.

Procedure for cleaning the conical flask:

- Clean with deionised water only. *[Any water droplets remaining won't change the number of moles of reactant you add].*

Acid/Base Titrations:

1. To standardise a solution of HCl using a standard solution of Na₂CO₃.

Acid: HCl – strong acid

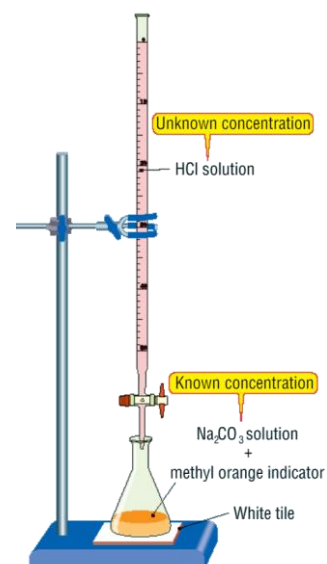
Base: Na₂CO₃ – weak base

Indicator: Methyl Orange (SAWBMO)

Colour change: Yellow to Red

Equation: $2\text{HCl} + \text{Na}_2\text{CO}_3 \rightarrow 2\text{NaCl} + \text{H}_2\text{O} + \text{CO}_2$

Ratio: 2 HCl : 1 Na₂CO₃



Sample calculation: (2012 HL Q1)

A student determined the concentration of a hydrochloric acid solution by titration with 25.0 cm³ portions of a 0.05 M primary standard solution of anhydrous sodium carbonate. The portions of sodium carbonate solution were measured into a conical flask using a 25 cm³ pipette. The hydrochloric acid solution was added from a burette. The mean titre was 20.8 cm³.

The balanced equation for the titration reaction was:



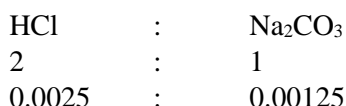
(e) Calculate, correct to two decimal places, the concentration of the hydrochloric acid solution in

(i) moles per litre,

(ii) grams per litre. (12)

- (i) We have 0.05 mol/L Na₂CO₃

$$\frac{0.05 \times 25}{1000}$$
 = 0.00125 mol/25 cm³ Na₂CO₃



$$\frac{0.0025 \text{ mol}/20.8 \text{ cm}^3 \text{ HCl}}{0.0025 \times 1000}$$

$$\frac{20.8}{20.8}$$

$$= 0.12 \text{ mol/L HCl}$$

- (ii) M_r of HCl = (1) + (35.5) = 36.5

$$0.12 \times 36.5 = 4.38 \text{ g/L HCl} \quad [\text{mol to g} \rightarrow \times M_r]$$

2. To determine the concentration of ethanoic acid in vinegar

Acid: CH₃COOH – weak acid

Base: NaOH – strong base

Indicator: Phenolphthalein (WASBPH)

Colour change: Pink to Colourless [NOT “clear”!]

Equation: CH₃COOH + NaOH → CH₃COONa + H₂O

Ratio: 1 CH₃COOH : 1 NaOH

Notes: Vinegar must be diluted beforehand because it is too concentrated. Make sure to multiply the concentration of the *dilute* vinegar by the dilution factor to find the concentration of the *original* vinegar.

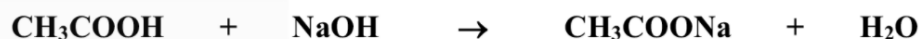
$$\text{Dilution factor} = \frac{\text{Volume of Diluted Vinegar}}{\text{Volume of Original Vinegar}}$$

Clear vinegar should be used to ensure the endpoint is clearly seen.

Sample calculation: (2016 HL Q1)

To determine the concentration of ethanoic acid in a sample of vinegar, 25.0 cm³ of the vinegar were diluted to 250 cm³ and then the diluted vinegar was titrated with a previously standardised solution which contained 1.20 g of sodium hydroxide in 500 cm³ of solution. On average, 18.75 cm³ of the diluted vinegar were required to neutralise 25.0 cm³ of this sodium hydroxide solution.

The equation for the titration reaction is:



(c) Calculate

- (i) the number of moles of sodium hydroxide in each 25.0 cm³ portion,
- (ii) the number of moles of ethanoic acid per cm³ of *diluted* vinegar. (12)

(d) Find the concentration of ethanoic acid in the *original* vinegar

- (i) in terms of moles per litre,
- (ii) as a percentage (w/v). (9)

(c)

- (i) We have 1.20 g/500 cm³ NaOH
 M_r of NaOH = (23) + (16) + (1) = 40
 $1.20 \div 40$ [g to mol $\rightarrow \div M_r$]
 = 0.03 mol/500 cm³ NaOH
 $\frac{0.03 \times 25}{500}$
 = 0.0015 mol/25 cm³ NaOH

- (ii)

CH ₃ COOH	:	NaOH
1	:	1
0.0015	:	0.0015

$$\begin{aligned} &0.0015 \text{ mol}/18.75 \text{ cm}^3 \text{ CH}_3\text{COOH (dilute)} \\ &\frac{0.0015 \times 1}{18.75} \\ &= 0.00008 \text{ mol}/1 \text{ cm}^3 \text{ CH}_3\text{COOH (dilute)} \end{aligned}$$

(d)

- (i)

$0.00008 \text{ mol}/1 \text{ cm}^3 \text{ CH}_3\text{COOH (dilute)}$ $\times 10$ $= 0.0008 \text{ mol}/1 \text{ cm}^3 \text{ CH}_3\text{COOH (original)}$ $\times 1000$ $= 0.8 \text{ mol/L CH}_3\text{COOH (original)}$	$\text{Dilution Factor} = \frac{250}{25} = 10$
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(ii) % (w/v) means g/100 cm³

We have 0.8 mol/L CH₃COOH (original)

× 60 [mol to g → × M_r]

= 48 g/L CH₃COOH (original)

÷ 10

= 4.8 g/100 cm³ CH₃COOH (original)

= 4.8 % (w/v)

M_r of CH₃COOH = (12) + 3(1) + (12) + (16) + (16) + (1) = 60

3. To determine the amount of water of crystallisation in washing soda (hydrated sodium carbonate)

Acid: HCl – strong acid

Base: Na₂CO₃ – weak base

Indicator: Methyl Orange (SAWBM0)

Colour change: Yellow to Red

Equation: 2HCl + Na₂CO₃ → NaCl + H₂O + CO₂

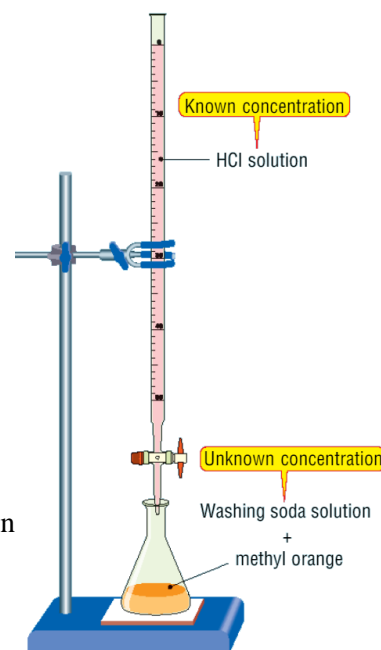
Ratio: 2 HCl : 1 Na₂CO₃

Notes: Same as titration number 1, just with extra calculations.

The crystals are made up of Na₂CO₃·xH₂O. We need to find 2 things:

- 1- the percentage of water of crystallisation, and
- 2- the value of x.

These calculations can also come up for other compounds – not just in in this experiment, e.g. finding the percentage water of crystallisation and value for x in hydrated copper (II) sulphate, CuSO₄·xH₂O.



Sample Calculation:

An experiment was carried out to determine the percentage water of crystallisation and the degree of water of crystallisation, **x**, in a sample of hydrated sodium carbonate crystals (Na₂CO₃·xH₂O). An 8.20 g sample of the crystals was weighed accurately on a clock glass and then made up to 500 cm³ of solution in a volumetric flask. A pipette was used to transfer 25.0 cm³ portions of this solution to a conical flask. A previously standardised 0.11 M hydrochloric acid (HCl) solution was used to titrate each sample. A number of accurate titrations were carried out. The average volume of hydrochloric acid solution required in these titrations was 26.05 cm³.

The titration reaction is described by the equation:



(d) From the titration figures, calculate the concentration of sodium carbonate (Na₂CO₃) in the solution in

(i) moles per litre,

(ii) grams per litre.

(9)

(e) Calculate the percentage water of crystallisation present in the crystals and the value of **x**, the degree of hydration of the crystals.

(12)

(d)

(i) We have 0.11 mol/L HCl

$$\frac{0.11 \times 26.05}{1000}$$

$$= 0.0028655 \text{ mol/26.05 cm}^3 \text{ HCl}$$

$$\begin{array}{ccc} \text{HCl} & : & \text{Na}_2\text{CO}_3 \\ 2 & : & 1 \\ 0.0028655 & : & 0.00143275 \end{array}$$

$$0.00143275 \text{ mol/25 cm}^3 \text{ Na}_2\text{CO}_3$$

$$\frac{0.00143275 \times 1000}{25}$$

$$= 0.05731 \text{ mol/L Na}_2\text{CO}_3$$

(ii) M_r of $\text{Na}_2\text{CO}_3 = 2(23) + (12) + 3(16) = 106$

$$\times 106 \quad [\text{mol to g} \rightarrow \times M_r]$$

$$= 6.07486 \text{ g/L Na}_2\text{CO}_3$$

(e)

(i) **Percentage water of crystallisation = $\frac{\text{Mass of water in your sample}}{\text{Mass of your sample}} \times 100$**

We had 6.07486 g/L Na_2CO_3 but we only made 500 cm^3 of the solution.

$$\div 2$$

$$= 3.03743 \text{ g/500 cm}^3 \text{ Na}_2\text{CO}_3 \text{ in our solution.}$$

But we weighed out 8.20g of crystals.

The extra mass is the mass of water.

$$8.20 - 3.03743$$

$$= 5.16257 \text{ g of water in our crystals}$$

$$\% \text{ water of crystallisation} = \frac{5.16257}{8.20} \times 100$$

(ii)

$$\frac{\text{Mass of Na}_2\text{CO}_3}{M_r \text{ of Na}_2\text{CO}_3} = \frac{\text{Mass of } x\text{H}_2\text{O}}{M_r \text{ of } x\text{H}_2\text{O}}$$

$$M_r \text{ of } x\text{H}_2\text{O} = x[2(1) + (16)] = 18x$$

$$\frac{3.03743}{106} = \frac{5.16257}{18x}$$

$$x = \frac{5.16257 \times 106}{18 \times 3.03743}$$

$$x = 10 \quad [\text{round to a whole number if possible}]$$

This means our crystals are actually made up of $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$