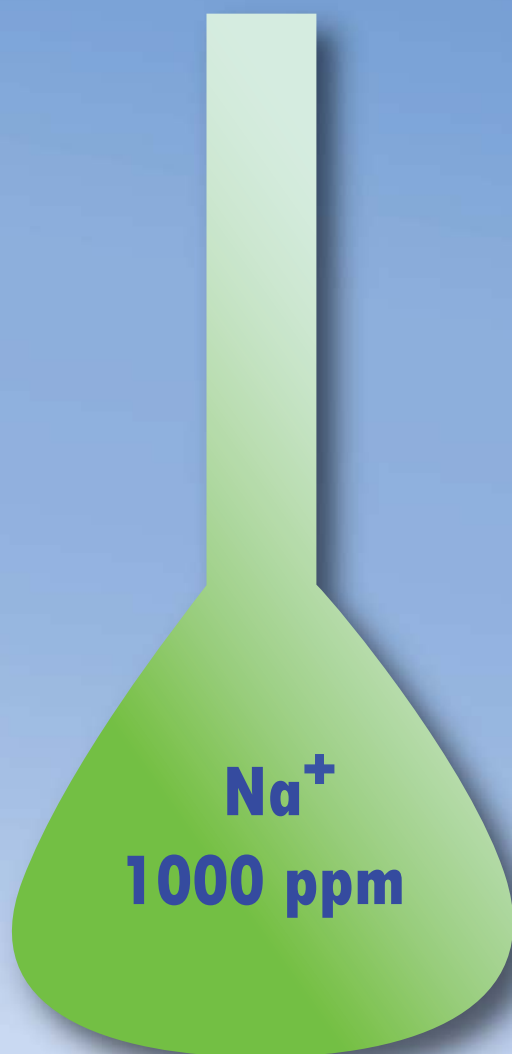


Guide To Preparation of Stock Standard Solutions

**First Edition
May 2011**



Guide To Preparation Of Stock Standard Solutions

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May – 2011

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Forward

We are looking to make chemistry simple, easy for student, teacher, chemist, analyst and scientist. We are trying our best starting from small principles in chemistry. We found some chemists are making some mistakes in calculation. This simple book is a practical training for chemist, technicians or scientist for solutions calculations.

Who Should Read it?

This help book is written to be readable by **anyone** who has idea in basic principles of general chemistry. It will help student, teachers, chemist, scientist and engineers ... etc who's working in chemistry field to fully understand the preparation of stock standard solution. It will start from basic calculations up to practical preparation procedure in lab.

Chapter one is an introduction explain basic principles of calculations for concentration units and conversion between them. It also considers view examples in each section and general example at the end of chapter. Including a lot of examples in each chapter is to help readers to fulfill understand principles in the preparation of standard solution.

Chapter two consider types of stock standards depending on the formula types of analyte, because of different applications in lab and different methods used in analysis, some by ICP for metals, titration or Ion Chromatography for anions and cations and Formula standards for preparation of reagents. Calculations for each type are slightly same but we make it in different sections to simplify the idea of stock standards preparation. Because some chemists still can't distinguish between them.

Chapter three include practical laboratory guide. It is include some basic principles and guide to work in lab for preparing stock standard solution.

Chapter 1: Introduction



1.1 Stock Standard Solution:

Analysis in any laboratory mainly based on reference materials like Stock standard solution. The accuracy in the preparation of stock standard reflects accuracy of the results. Stock standard solution is defined as a solution with high concentration of stable analyte(s) that can be stored at specific conditions in laboratory for long time and used as a standard reference material for analysis of the target analyte(s) in the daily use. Example of stock standard solutions for like Sodium (Na) solution with a concentration of 1000 mg Na /L (ppm) used as stock solution for Sodium analysis by Inductively Coupled Plasma (ICP), Flame Photometry, Ion Chromatography (IC)...etc, that can be stored at 4 °C for a minimum of 6 months. All stock standards shall be checked before use with another standard that has been prepared separately from different source.

To prepare stock standard solution in lab you should first make sure that you understand the concentration unit principles and some mathematical rules which will help to find exact answer for reporting.

1.2 Scientific Notation

Scientific notation is simply a method for expressing, and working with, very large or very small numbers. It is a short hand method for writing numbers, and an easy method for calculations. Numbers in scientific notation are made up of three parts: the coefficient, the base and the exponent. Scientific notation expression:

$$N \times 10^n$$

Where ($1 < N < 10$)

Example number 2,890,000,000 becomes 2.89×10^9 and 0.0000672 expressed in scientific notation as 6.72×10^{-5}

Good Practice: Express the following numbers in scientific notation: (a) 0.0000073 (b) 344.5 (c) 12,3421.0

1.3 Significant Figures

Every experiment (except in some counting situations) involves a degree of uncertainty. Thus, suppose that several people measure the length of a sheet of paper, using a ruler that is divided into tenths of a centimeter, and get the following results: 27.92 cm, 27.96 cm, 27.90 cm, 2.10 cm. Notice that everyone

agrees as to the first three digits (except the last person who is measuring from the wrong end of a 30 cm long ruler!). Clearly the fourth digit, which has been estimated by everyone, is a doubtful figure (in fact even the third figure may be doubtful if the ruler is too short or too long due to manufacturing defect or environmental factors).

Definition: The digits that are considered correct and the first doubtful digit are called significant figures.

The number of significant figures in a measurement depends upon the precision of the instrument and, to some extent, upon the skill of the measurer. An effort should always be made to obtain as many figures as an instrument will allow. Conversely, only significant figures should be recorded in taking data – i.e., a measurement should not be written in such a way as to imply a greater precision than is actually inherent in the measuring device and/or measuring technique.

For example, if we weigh a sample on a balance and record its mass as 1.2637 g, we assume that all digits, except the last, are known exactly. We assume that the last digit has an uncertainty of at least ± 1 , giving an absolute uncertainty of at least ± 0.0001 g, or a relative uncertainty of at least

$$\frac{\pm 0.0001 \text{ g}}{1.2637 \text{ g}} \times 100 = \pm 0.0079 \%$$

Significant figures are a reflection of a measurement's uncertainty. The number of significant figures is equal to the number of digits in the measurement, with the exception that a zero (0) used to fix the location of a decimal point is not considered significant. This definition can be ambiguous. For example, how many significant figures are in the number 100? If measured to the nearest hundred, then there is one significant figure. If measured to the nearest ten, however, then two significant figures are included. To avoid ambiguity we use scientific notation. Thus, 1×10^2 has one significant figure, whereas 1.0×10^2 has two significant figures.

For measurements using logarithms, such as pH, the number of significant figures is equal to the number of digits to the right of the decimal, including all zeros. Digits to the left of the decimal are not included as significant figures since they only indicate the power of 10. A pH of 2.45, therefore, contains two significant figures.

Exact numbers, such as the stoichiometric coefficients in a chemical formula or reaction, and unit conversion factors, have an infinite number of significant figures. A mole of CaCl_2 , for example, contains exactly two moles of chloride and one mole of calcium. In the equality

$$1000 \text{ mL} = 1 \text{ L}$$

both numbers have an infinite number of significant figures.

Recording a measurement to the correct number of significant figures is important because it tells others about how precisely you made your measurement. For example, suppose you weigh an object on a balance capable of measuring mass to the nearest ± 0.1 mg, but record its mass as 1.762 g instead of 1.7620 g. By failing to record the trailing zero, which is a significant figure, you suggest to others that the mass was determined using a balance capable of weighing to only the nearest ± 1 mg. Similarly, a buret with scale markings every 0.1 mL can be read to the nearest ± 0.01 mL. The digit in the hundredth's place is the least significant figure since we must estimate its value. Reporting a volume of 12.241 mL implies that your buret's scale is more precise than it actually is, with divisions every 0.01 mL.

Significant figures are also important because they guide us in reporting the result of an analysis. When using a measurement in a calculation, the result of that calculation can never be more certain than that measurement's uncertainty. Simply put, the result of an analysis can never be more certain than the least certain measurement included in the analysis.

Finding the number of significant figures in a measurement is usually easy but can be troublesome if zeros are present. Look at the following four quantities:

4.803 cm Four significant figures: 4, 8, 0, 3

0.006 61 g Three significant figures: 6, 6, 1

55.220 K Five significant figures: 5, 5, 2, 2, 0

34,200 m Anywhere from three (3, 4, 2) to five (3, 4, 2, 0, 0) significant figures

The following rules cover the different situations that can arise:

1. Zeros in the middle of a number are like any other digit; they are always significant. Thus, 4.803 cm has four significant figures.
2. Zeros at the beginning of a number are not significant; they act only to locate the decimal point. Thus, 0.00661 g has three significant figures. (Note that 0.00661 g can be rewritten as 6.61×10^{-3} or as 6.61 mg.)
3. Zeros at the end of a number and after the decimal point are always significant. The assumption is that these zeros would not be shown unless they were significant. Thus, 55.220 K has five significant figures. (If the value were known to only four significant figures, we would write 55.22 K.)
4. Zeros at the end of a number and before the decimal point may or may not be significant. We can't tell whether they are part of the measurement or whether they only locate the decimal point. Thus, 34,200 m may have three, four, or five significant figures. Often, however, a little common sense is helpful. A temperature reading of 20°C probably has two significant figures

rather than one, since one significant figure would imply a temperature anywhere from 10–30°C and would be of little use. Similarly, a volume given as 300 mL probably has three significant figures. On the other hand, a figure of 93,000,000 mi for the distance between the earth and the sun probably has only two or three significant figures.

The fourth rule shows why it's helpful to write numbers in scientific notation rather than ordinary notation. Doing so makes it possible to indicate the number of significant figures. Thus, writing the number 34,200 as 3.42×10^4 indicates three significant figures, but writing it as 3.4200×10^4 indicates five significant figures.

One further point about significant figures: Certain numbers, such as those obtained when counting objects, are exact and have an effectively infinite number of significant figures. For example, a week has exactly 7 days, not 6.9 or 7.0 or 7.1, and a foot has exactly 12 in., not 11.9 or 12.0 or 12.1. In addition, the power of 10 used in scientific notation is an exact number. That is, the number is exact, but the number has one significant figure.

Example 1.1

How many significant figures in each of the following numbers:

- (a) 6.07×10^{-15}
- (b) 0.003840
- (c) 463.8052

SOLUTION

- (a) Three significant figures 6, 0, 5
- (b) Four significant figures 3, 8, 4, 0
- (c) Seven significant figures 4, 6, 3, 8, 0, 5, 2

1.3.1 Addition and Subtraction of Significant Figures

For the addition and subtraction the result has the same number of decimal places as the least precise measurement used in the calculation.

For example: $98.112 + 2.3 = 100.412$ but this value must be rounded to 100.4

Example 1.2

Calculate each following mathematical operation to correct number of significant figures:

(a) $212.2 + 26.7 + 402.09$

(b) $1.0028 - 0.221$

SOLUTION

For addition and subtraction final calculated value rounded the lowest number of decimal places:

- (a) $212.2 + 26.7 + 402.09$, decimal places are 1, 1, 2 respectively and after calculation the final result should contain one decimal places only

$$212.2 + 26.7 + 402.09 = 640.49 \text{ rounded to one decimal places become} \\ = 640.5$$

- (b) $1.0028 - 0.221$, decimal places are 4, 3 respectively and final result should have only 3 decimal places

$$1.0028 - 0.221 = 0.7818 \text{ become after rounding} \\ = 0.782$$

1.3.2 Multiplication and Division of Significant Figures

When multiplying, dividing, or taking roots, the result should have the same number of significant figures as the least precise number in the calculation.

$(3.69)(2.3059) = 8.5088$, which should be rounded to 8.51 (three significant figures like 3.69)

Example 1.3

Perform the following calculation to correct number of significant figures:

(a) 0.0821×273

(b) $7.89 \div 2.4$

SOLUTION

- (a) 0.08255×273 , number of significant figures for both numbers is 3, 4 respectively then final result should contain only three.

$$0.8255 \times 273 = 22.53615 \longrightarrow \text{rounding to 3 significant figures} \\ = 22.5$$

- (b) $7.89 \div 2.4$, number of significant figures for both numbers is 3, 2 respectively then final result should contain only two.

$$7.89 \div 2.4 = 3.2875 \longrightarrow \text{rounding to 2 significant figures} \\ = 3.3$$

1.3.3 Mathematical Operation

Example 1.4

Carry out the following mathematical operations, and give each result with the correct number of significant figures.

- $1.05 \times 10^{-3} \div 6.135$
- $21 - 13.8$
- As part of a lab assignment to determine the value of the gas constant (R), a student measured the pressure (P), volume (V), and temperature (T) for a sample of gas, where

$$R = \frac{PV}{T}$$

The following values were obtained: $P = 2.560$, $T = 275.15$, and $V = 8.8$. (Gases will be discussed in detail in Chapter 5; we will not be concerned at this time about the units for these quantities.) Calculate R to the correct number of significant figures.

- The result is 1.71×10^{-4} , which has three significant figures because the term with the least precision (1.05×10^{-3}) has three significant figures.
- The result is 7 with no decimal point because the number with the least number of decimal places (21) has none.
- $R = \frac{PV}{T} = \frac{(2.560)(8.8)}{275.15}$

The correct procedure for obtaining the final result can be represented as follows:

$$\frac{(2.560)(8.8)}{275.15} = \frac{22.528}{275.15} = 0.0818753 \\ = 0.082 = 8.2 \times 10^{-2} = R$$

The final result must be rounded to two significant figures because 8.8 (the least precise measurement) has two significant figures. To show the effects of rounding at intermediate steps, we will carry out the calculation as follows:

$$\begin{array}{c}
 \text{Rounded to two} \\
 \text{significant figures} \\
 \downarrow \\
 \frac{(2.560)(8.8)}{275.15} = \frac{22.528}{275.15} = \frac{23}{275.15}
 \end{array}$$

Now we proceed with the next calculation:

$$\frac{23}{275.15} = 0.0835908$$

Rounded to two significant figures, this result is:

$$0.084 = 8.4 \times 10^{-2}$$

Note that intermediate rounding gives a significantly different result than was obtained by rounding only at the end. Again, we must reemphasize that in your calculations you should round only at the end.

However, because rounding is carried out at intermediate steps in this text (to always show the correct number of significant figures), the final answer given in the text may differ slightly from the one you obtain (rounding only at the end).

There is a useful lesson to be learned from part c of above exercise. The student measured the pressure and temperature to greater precision than the volume. A more precise value of R (one with more significant figures) could have been obtained if a more precise measurement of V had been made. As it is, the efforts expended to measure P and T very precisely were wasted. Remember that a series of measurements to obtain some final result should all be done to about the same precision.

1.4 Rounding Result

When a value contains too many significant figures, it must be rounded off. Rounding method involves underestimating the value when rounding the five digits 0, 1, 2, 3, and 4, and overestimating the value when rounding the five digits 5, 6, 7, 8, and 9. With this approach, if the value of the digit to the right of the last significant figure is smaller than 5, drop this digit and leave the remaining number unchanged. Thus, 2.794 becomes 2.79. If the value of the digit to the right of the last significant digit is 5 or larger, drop this digit and add 1 to the preceding digit. Thus, 2.795 become 2.80.

In chemistry there are several rules or bases to round final result of calculations:

- Rounding to correct number of significant figures which are mostly applicable for laboratory reported final results
- Rounding result to specific number of decimal places, 1, 2, 3, 4 or more depend upon the precision or calculation purpose we would like to reach

Unless specified otherwise all this book examples we will follow second rule of rounding final results, because this book prepared to help analyst to find the exact weight and most analytical balances are prepared to weigh up to four decimal places (0.0000 ± 0.0001).

1.5 Expressing Concentration of Solute:

Concentration is a general measurement unit stating the amount of solute present in a known amount of solution:

$$\text{Concentration} = \frac{\text{Amount of Solute}}{\text{Amount of Solution}}$$

Although the terms “Solute” and “Solution” are often associated with liquid samples, they can be extended to gas-phase and solid-phase samples as well. The actual units for reporting concentration depend on how the amount of solute and solution are measured. Table 1.1 shows the most common practical units used for expressing the concentration of solute.

Table 1.1: Common Practical Units for Reporting Concentration

Name	Units	Symbol
Molarity	$\frac{\text{moles of solute}}{\text{liters of solution}}$	M
Formality	$\frac{\text{moles of FWs solute}}{\text{liters of solution}}$	F
Normality	$\frac{\text{Number of EWs solute}}{\text{Liters of solution}}$	N
molality	$\frac{\text{moles of solute}}{\text{Kg of solvent}}$	m
Weight %	$\frac{\text{g of solute}}{100 \text{ g of solution}}$	% w/w
Volume %	$\frac{\text{mL of solute}}{100 \text{ mL of solution}}$	% v/v
Weight-to-Volume %	$\frac{\text{g of solute}}{100 \text{ mL of solution}}$	% w/v

1.5.1 Molarity and Formality

Both molarity and formality express concentration as moles of solute per liter of solution. There is, however, a subtle difference between molarity and formality. **Molarity** is the concentration of a particular chemical species in solution. **Formality**, on the other hand, is a substance's total concentration in solution without regard to its specific chemical form. There is no difference between a substance's molarity and formality if it dissolves without dissociating into ions. The molar concentration of a solution of glucose, for example, is the same as its formality.

For substances that ionize in solution, such as NaCl, molarity and formality are different. For example, dissolving 0.1 mol of NaCl in 1 L of water gives a solution containing 0.1 mol of Na^+ and 0.1 mol of Cl^- . The molarity of NaCl, therefore, is zero since there is essentially no undissociated NaCl in solution. The solution, instead, is 0.1 M in Na^+ and 0.1 M in Cl^- . The formality of NaCl, however, is 0.1 F because it represents the total amount of NaCl in solution. The rigorous definition of molarity, for better or worse, is largely ignored in the current literature, as it is in this text. When we state that a solution is 0.1 M NaCl we understand it to consist of Na^+ and Cl^- ions. The unit of formality is used only when it provides a clearer description of solution chemistry.

Molar concentrations are used so frequently that a symbolic notation is often used to simplify its expression in equations and writing. The use of square brackets around a species indicates that we are referring to that species' molar concentration. Thus, $[\text{Na}^+]$ is read as the “molar concentration of sodium ions”.

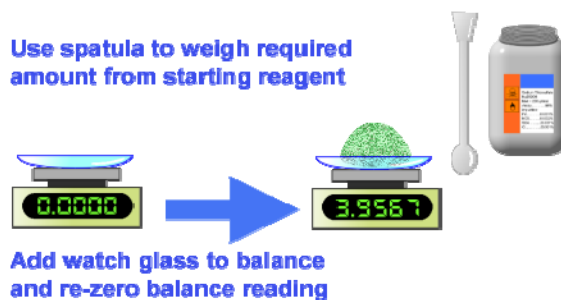
$$\text{Molarity} = \frac{\text{Moles of solute}}{\text{Volume of solution (L)}}$$

$$\text{Moles of solute} = \text{Molarity} \times \text{Volume of solution (L)}$$

$$\text{Volume of solution} = \frac{\text{Moles of solute}}{\text{Molarity}}$$

$$\text{Moles of solute} = \frac{\text{Weight of solute (g)}}{\text{Molecular Weight (g/mol)}}$$

Note that it is the final volume of the solution that is important, not the starting volume of the solvent used. The final volume of the solution might be a bit larger than the volume of the solvent because of the additional volume of the solute. In practice, a solution of known molarity is prepared by weighing an appropriate amount of solute and placing it in a volumetric flask. Enough solvent is added to dissolve the solute, and further solvent is added until an accurately calibrated final volume is reached. The solution is then shaken until it is uniformly mixed as show in figure 1.1.



(a)

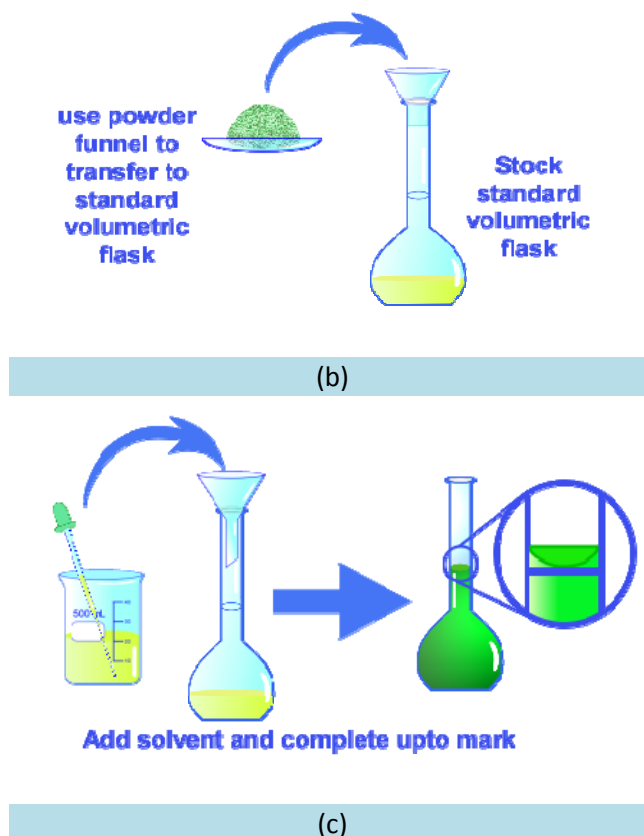


Figure 1.1: Preparing a stock solution of known molarity. (a) A measured number of moles of solute is weighed using analytical balance. (b) Solute is transferred in a volumetric flask. (c) Enough solvent is added to dissolve the solute by swirling and further solvent is carefully added until the calibration mark on the neck of the flask is reached, and the solution is then shaken until uniform.

Molarity can be used as a conversion factor to relate a solution's volume to the number of moles of solute. If we know the molarity and volume of a solution, we can calculate the number of moles of solute. If we know the number of moles of solute and the molarity of the solution, we can find the solution's volume. Worked

Examples 1.1 and 1.2 show how such calculations are done.

Example 1.1

What is the molarity of a solution made by dissolving 2.355 g of sulfuric acid in water and diluting to a final volume of 50.0 mL?

SOLUTION

Molarity is the number of moles of solute per liter of solution. Thus it's necessary to find the number of moles of sulfuric acid in 2.355 g and then divide by the volume of the solution.

Molar mass of $\text{H}_2\text{SO}_4 = (2 \times 1.0 \text{ g/mol}) + (1 \times 32.1 \text{ g/mol}) + (4 \times 16.0 \text{ g/mol}) = 98.1 \text{ g/mol}$

$$2.355 \text{ g } \cancel{\text{H}_2\text{SO}_4} \times \frac{1 \text{ mol } \text{H}_2\text{SO}_4}{98.1 \text{ g } \cancel{\text{H}_2\text{SO}_4}} = 0.0240 \text{ mol } \text{H}_2\text{SO}_4$$

$$\text{Molarity} = \frac{\text{Moles of solute}}{\text{Volume of solution (L)}} = \frac{0.0240 \text{ mol H}_2\text{SO}_4}{0.0500 \text{ L}} = 0.480 \text{ M}$$

The solution has a sulfuric acid concentration of 0.480 M

Example 1.2

Hydrochloric acid is sold commercially as a 12.0 M solution. How many moles of HCl are in 300.0 mL of 12.0 M solution?

SOLUTION

The number of moles of solute is calculated by multiplying the molarity of the solution by its volume.

Moles of HCl = (Molarity of solution) × (Volume of solution L)

$$= \frac{12.0 \text{ mol HCl}}{1 \cancel{\text{L Solution}}} \times 0.300 \cancel{\text{L}} = 3.60 \text{ mol HCl}$$

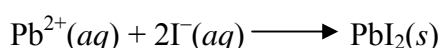
There are 3.60 mol of HCl in 300.0 mL of 12.0 M solution.

1.5.2 Normality

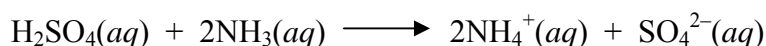
Normality is an older unit of concentration that, although once commonly used, is frequently ignored in today's laboratories. Normality is still used in some handbooks of analytical methods, and, for this reason, it is helpful to understand its meaning. For example, normality is the concentration unit used in *Standard Methods for the Examination of Water and Wastewater*, and in some *Standard EPA methods* commonly used sources of analytical methods for environmental laboratories.

Normality makes use of the chemical equivalent, which is the amount of one chemical species reacting stoichiometrically with another chemical species. Note that this definition makes an equivalent, and thus normality, a function of the chemical reaction in which the species participates. Although a solution of H₂SO₄ has a fixed molarity, its normality depends on how it reacts.

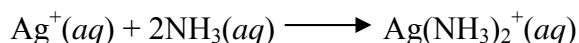
The number of **equivalents**, *n*, is based on a reaction unit, which is that part of a chemical species involved in a reaction. In a precipitation reaction, for example, the reaction unit is the charge of the cation or anion involved in the reaction; thus for the reaction



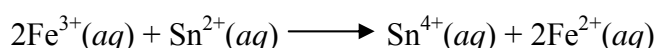
n = 2 for Pb²⁺ because each ion takes two electrons and *n* = 1 for I[−] because each ion donate only one electron. In an acid–base reaction, the reaction unit is the number of H⁺ ions donated by an acid or accepted by a base. For the reaction between sulfuric acid and ammonia



we find that $n = 2$ for H_2SO_4 because each molecule donate two ions of H^+ and $n = 1$ for NH_3 because each ion accept one H^+ . For a complexation reaction, the reaction unit is the number of electron pairs that can be accepted by the metal or donated by the ligand. In the reaction between Ag^+ and NH_3



the value of n for Ag^+ is 2 because each ion accept pair of electrons in covalent bonds with Ammonia NH_3 one electron from each covalent bond, for NH_3 is $n = 1$ because each molecule of ammonia donate one electron in each covalent bond it form with Ag^+ . Finally, in an oxidation–reduction reaction the reaction unit is the number of electrons released by the reducing agent or accepted by the oxidizing agent; thus, for the reaction



$n = 1$ for Fe^{3+} because each ion accept one electron in the reduction step and $n = 2$ for Sn^{2+} because each ion donate two electrons in the oxidation step. Clearly, determining the number of equivalents for a chemical species requires an understanding of how it reacts.

Normality is the number of **equivalent weights** (EW) per unit volume and, like formality, is independent of speciation. An equivalent weight is defined as the ratio of a chemical species' **formula weight** (FW) to the number of its equivalents

$$\text{Normality} = \frac{\text{Number of EWs solute}}{\text{liters of solution}}$$

$$\text{Number of EWs solute} = \frac{\text{Weight of solute}}{\text{Equivalent Weight (EW)}}$$

$$\text{Equivalent Weight (EW)} = \frac{\text{Formula Weight (g/mol)}}{n}$$

$$\text{Normality} = \frac{\text{Weight of solute}}{\text{Formula Weight} \times \text{liters of solution}} \times n$$

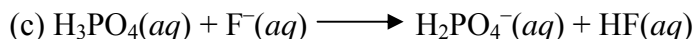
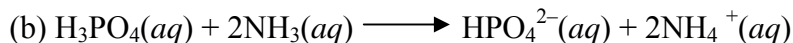
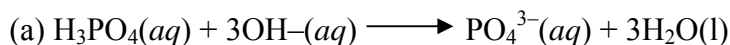
Consequently, the following simple relationship exists between normality and molarity.

$$N = n \times M$$

This equation is the simple form to fully understand the normality, you have to be able to determine the number of equivalents and calculate the molarity then use above equation to calculate the normality of the target analyte. Example 1.1 illustrates the relationship among chemical reactivity, equivalent weight, and normality.

Example 1.3

Calculate the equivalent weight and normality for a solution of 6.0 M H_3PO_4 given the following reactions:

**SOLUTION**

For phosphoric acid, the number of equivalents is the number of H^+ ion donated to the base. For the reactions in (a), (b), and (c) the number of equivalents are 3, 2, and 1, respectively. Thus, the calculated equivalent weights and normalities are

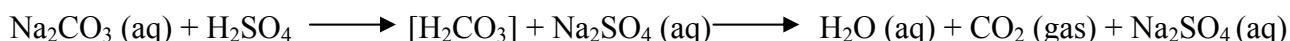
$$(a) \text{EW} = \frac{\text{FW}}{n} = \frac{97.994}{3} = 23.665 \quad N = n \times M = 3 \times 6.0 = 18.0 \text{ N}$$

$$(b) \text{EW} = \frac{\text{FW}}{n} = \frac{97.994}{2} = 48.997 \quad N = n \times M = 2 \times 6.0 = 12.0 \text{ N}$$

$$(c) \text{EW} = \frac{\text{FW}}{n} = \frac{97.994}{1} = 97.994 \quad N = n \times M = 1 \times 6.0 = 6.0 \text{ N}$$

Example 1.4

In standard method for Alkalinity measurement, a solution of 0.05N of Na_2CO_3 should be prepared based on the following titration reaction with sulfuric acid:



The part $[\text{H}_2\text{CO}_3]$ is an intermediate which is directly converted to H_2O and CO_2 gas.

How many grams of Na_2CO_3 required to prepare 1.0 Liter of 0.05N solution?

SOLUTION 1 By Normality Equations:

According to the reaction above (acid-base reaction), each molecule of Na_2CO_3 accept two hydrogen ions (H^+) from sulfuric acid, then number of equivalents for Na_2CO_3 $n=2$.

Formula Weight (FW) for $\text{Na}_2\text{CO}_3 = 105.99 \text{ g/mol}$

$$\text{Equivalents Weight EW} = \frac{\text{FW}}{n} = \frac{105.99}{2} = 52.995 \text{ g/mol}$$

$$\text{Normality} = \frac{\text{Number of EWs solute}}{\text{liters of solution}} \longrightarrow \text{Number of EWs solute} = \text{Normality} \times \text{Liter of solution}$$

$$\text{Number of EWs solute} = 0.05 \text{ N} \times 1.0 \text{ L} = 0.05 \text{ mol}$$

$$\text{Number of EWs solute} = \frac{\text{Weight of solute}}{\text{Equivalent Weight (EW)}} \longrightarrow$$

$$\text{Weight of solute} = \text{Number of EWs solute} \times \text{Equivalent Weight (EW)} = 0.05 \text{ mol} \times 52.995 \text{ g/mol} = 2.65 \text{ g}$$

To prepare 0.05N of Na_2CO_3 weigh 2.65 g of Na_2CO_3 and dissolve and complete to volume 1.0 L.

SOLUTION 2 By Molarity Equations:

Convert the normality concentration to molarity the make all calculations by molarity equations

$$N = n \times M \longrightarrow M = \frac{N}{n} = \frac{0.05}{2} = 0.025 \text{ M of } \text{Na}_2\text{CO}_3$$

$$\text{Moles of solute} = \text{Molarity} \times \text{Volume of solution (L)}$$

$$\text{Moles of solute} = 0.025 \text{ M} \times 1.0 \text{ L} = 0.025 \text{ mol of } \text{Na}_2\text{CO}_3$$

$$\text{Moles of solute} = \frac{\text{Weight of solute (g)}}{\text{Molecular Weight (g/mol)}} \longrightarrow$$

$$\text{Weight of solute (g)} = \text{Moles of solute} \times \text{Molecular Weight (g/mol)}$$

$$\text{Weight of solute (g)} = 0.025 \text{ mol} \times 105.99 \text{ (g/mol)} = 2.65 \text{ g of } \text{Na}_2\text{CO}_3$$

1.5.3 Molality

Molality is used in thermodynamic calculations where a temperature independent unit of concentration is needed. Molarity, formality and normality are based on the volume of solution in which the solute is dissolved. Since density is a temperature dependent property a solution's volume, and thus it's molar, formal and normal concentrations, will change as a function of its temperature. By using the solvent's mass in place of its volume, the resulting concentration becomes independent of temperature.

Molality is defined as mole of solute in Kg of solvent as per equation:

$$\text{molality} = \frac{\text{moles of solute}}{\text{Kg of solvent}}$$

Example 1.5

What is the molality of solution made by dissolve 25 g of NaCl in to 2.0 Liter of water. Assume the density of water $d = 1.0 \text{ g/mL} (= \text{kg/L})$.

SOLUTION

$$\text{Molar mass of NaCl} = (1 \times 22.99 \text{ g/mol}) + (1 \times 35.45 \text{ g/mol}) = 58.44 \text{ g}$$

$$25 \text{ g } \cancel{\text{NaCl}} \times \frac{1 \text{ mol NaCl}}{58.44 \text{ g } \cancel{\text{NaCl}}} = 0.428 \text{ mol of NaCl}$$

$$2.0 \cancel{\text{ Liter water}} \times \frac{1.0 \text{ kg}}{1.0 \cancel{\text{ Liter water}}} = 2.0 \text{ kg}$$

$$\text{molality} = \frac{\text{moles of solute}}{\text{Kg of solvent}} = \frac{0.428 \text{ mol NaCl}}{2.0 \text{ kg water}} = 0.214 \text{ m of NaCl}$$

The solution has concentration of NaCl equals to 0.214 m.

1.5.4 Weight, Volume, and Weight-to-Volume Ratios

Weight percent (% w/w), **volume percent** (% v/v) and **weight-to-volume percent** (% w/v) express concentration as units of solute per 100 units of solution. A solution in which a solute has a concentration of 23% w/v contains 23 g of solute per 100 mL of solution. **Parts per million** (ppm) and **parts per billion** (ppb) are mass ratios of grams of solute to one million or one billion grams of sample, respectively. For example, a steel that is 450 ppm in Mn contains 450 mg of Mn for every kilogram of steel. If we approximate the density of an aqueous solution as 1.00 g/mL, then solution concentrations can be expressed in parts per million or parts per billion using the following relationships. For gases a part per million usually is a volume ratio. Thus, a helium concentration of 6.3 ppm means that one liter of air contains 6.3 mL of He.

Table 1.2 explain most common equations used in calculations of weight and volume ratios units. Note that the ppm of ppb units can be used as (w/w) or (w/v), for solutions if unit is not specified as (w/v) or (w/w) then (w/v) is assumed as default.

Table 1.2: most common equations used in calculations of weight and volume ratios units.

Concentration Unit	Equations
% w/w	$= \frac{\text{g of solute}}{100 \text{ g of Solution}} = \frac{\text{g of solute}}{\text{g of Solution}} \times 100 \%$
%v/v	$= \frac{\text{mL of solute}}{100 \text{ mL of Solution}} = \frac{\text{mL of solute}}{\text{mL of Solution}} \times 100 \%$
%w/v	$= \frac{\text{g of solute}}{100 \text{ mL of Solution}} = \frac{\text{g of solute}}{\text{mL of Solution}} \times 100 \%$
Part per million (ppm) w/v	$= \frac{\text{mg of solute}}{\text{L of Solution}} = \frac{\mu\text{g of solute}}{\text{mL of Solution}} = \frac{\text{ng of solute}}{\mu\text{L of Solution}}$
Part per million (ppm) w/w	$= \frac{\text{mg of solute}}{\text{kg of Solution}} = \frac{\mu\text{g of solute}}{\text{g of Solution}} = \frac{\text{ng of solute}}{\text{mg of Solution}}$
Part per billion (ppb) w/v	$= \frac{\mu\text{g of solute}}{\text{L of Solution}} = \frac{\text{ng of solute}}{\text{mL of Solution}} = \frac{\text{pg of solute}}{\mu\text{L of Solution}}$

Concentration Unit	Equations
Part per billion (ppb) w/w	$= \frac{\mu\text{g of solute}}{\text{kg of Solution}} = \frac{\text{ng of solute}}{\text{g of Solution}} = \frac{\text{pg of solute}}{\text{mg of Solution}}$

w/w: Weight by weight

w/v: Weight by volume

v/v: Volume by volume

Example 1.6

How many grams of NaCl required to prepare each of the following solutions:

a) 2500 ppm (w/v) NaCl 250 mL solution.

b) 10% (w/v) NaCl in 250 mL solution.

c) 20% (w/w) NaCl in 250 g solution.

SOLUTION

a) 2500 ppm (w/v) NaCl

$$\text{Conc. (ppm)} = \frac{\text{mg of solute}}{\text{Volume of Solution (L)}} \longrightarrow \text{mg of solute} = \text{Conce. (ppm)} \times \text{Volume of Solution (L)}$$

$$\text{mg of NaCl} = 2500 \text{ ppm} \times 0.250 \text{ L} = 625 \text{ mg} = 0.625 \text{ g of NaCl.}$$

b) 10% (w/v) NaCl

$$\text{Conc. \% (w/v)} = \frac{\text{g of solute}}{\text{mL of Solution}} \times 100 \% \longrightarrow \text{g of solute} = \frac{\text{Conc. \% (w/v)} \times \text{mL of Solution}}{100 \%}$$

$$\text{g of NaCl} = \frac{10 \% (\text{w/v}) \times 250 \text{ mL Solution}}{100 \%} = 25 \text{ g}$$

c) 20% (w/w) NaCl

$$\text{Conc. \% (w/w)} = \frac{\text{g of solute}}{\text{g of Solution}} \times 100 \% \longrightarrow \text{g of solute} = \frac{\text{Conc. \% (w/w)} \times \text{g of Solution}}{100 \%}$$

$$\text{g of solute (NaCl)} = \frac{20 \% (\text{w/w}) \times 250 \text{ g of Solution}}{100 \%} = 50 \text{ g}$$

Example 1.7

What is the concentration of MgSO_4 the following prepared solution, express concentrations in ppm, % (w/v) and (w/w) concentrations units. Assume solution density is 1.0 g/mL

30 g of MgSO_4 dissolved in 500 mL distilled water.

SOLUTION

$$\text{Conc. (ppm)} = \frac{\text{mg of solute}}{\text{Volume of Solution (L)}} = \frac{30,000 \text{ mg MgSO}_4}{0.500 \text{ L}} = 60,000 \text{ ppm MgSO}_4$$

$$\text{Conc. \% (w/v)} = \frac{\text{g of solute}}{\text{mL of Solution}} \times 100 \% = \frac{30 \text{ g MgSO}_4}{500 \text{ mL}} \times 100 \% = 6 \% \text{ (w/v) MgSO}_4$$

When solution density 1.0 g/mL then

$$500 \text{ mL solution} \times \frac{1.0 \text{ g}}{1 \text{ mL Solution}} = 500 \text{ g}$$

$$\text{Conc. \% (w/w)} = \frac{\text{g of solute}}{\text{g of Solution}} \times 100 \% = \frac{30 \text{ g MgSO}_4}{500 \text{ g Solution}} \times 100 \% = 6 \% \text{ (w/w) MgSO}_4$$

1.6 Converting Between Concentration Units

Many scientific activities involve numerical calculations—measuring, weighing, preparing solutions, and so forth—and it's often necessary to convert a quantity from one unit to another. Converting between units isn't difficult; we all do it every day. If you run 7.5 laps around a 200 meter track, for instance, you have to

convert between the distance unit lap and the distance unit meter to find that you have run 1500 m (7.5 laps times 200 meters/lap). Converting from one scientific unit to another is just as easy.

$$7.5 \text{ Laps} \times \frac{200 \text{ meters}}{1 \text{ Lap}} = 1500 \text{ meters}$$

The simplest way to carry out calculations that involve different units is to use the dimensional-analysis method. In this method, a quantity described in one unit is converted into an equivalent quantity with a different unit by using a conversion factor to express the relationship between units:

$$\text{Original quantity} \times \text{Conversion factor} = \text{Equivalent quantity}$$

As an example, we know that 1 ppm equals 1000 ppb. Writing this relationship as a fraction restates it in the form of a conversion factor, either ppm per ppb or ppb per ppm:

$$\text{Conversion factor between ppm and ppb} \longrightarrow \frac{1 \text{ ppm}}{1000 \text{ ppb}} \text{ equals to } \frac{1000 \text{ ppb}}{1 \text{ ppm}} \text{ equals } 1$$

Note that this and all other conversion factors are numerically equal to 1 because the value of the quantity above the division line (the numerator) is equal to the value of the quantity below the division line (the denominator). Thus, multiplying by a conversion factor is equivalent to multiplying by 1 and so does not change the value of the quantity.

The key to the dimensional-analysis method of problem solving is that units are treated like numbers and can thus be multiplied and divided (though not added or subtracted) just as numbers can. The idea when solving a problem is to set up an equation so that unwanted units cancel, leaving only the desired units.

Usually it's best to start by writing what you know and then manipulating that known quantity. For example, if you know the volume is 153 mL and want to find it in deciliter (dL), you can write down the volume in mL and set up an equation multiplying the mL by the conversion factor in mL per dL:

$$153 \cancel{\text{mL}} \times \frac{1 \text{ dL}}{100 \cancel{\text{mL}}} = 1.53 \text{ dL}$$

The unit "mL" cancels from the left side of the equation because it appears both above and below the division line, and the only unit that remains is "dL". The dimensional-analysis method gives the right answer only if the equation is set up so that the unwanted units cancel. If the equation is set up in any other way, the units won't cancel properly, and you won't get the right answer. Thus, if you were to multiply volume in mL by the incorrect conversion factor mL per dL, you would end up with an incorrect answer expressed in meaningless units:

$$153 \text{ mL} \times \frac{100 \text{ mL}}{1 \text{ dL}} = 15300 \text{ mL}^2/\text{dL} \quad ??$$

The main drawback to using the dimensional-analysis method is that it's easy to get the right answer without really understanding what you're doing. It's therefore best after solving a problem to think through a rough estimate, or "ballpark" solution, as a check on your work. If your ballpark check isn't close to the detailed solution, there's a misunderstanding somewhere, and you should think the problem through again. Even if you don't make an estimate, it's important to be sure that your calculated answer makes sense. The dimensional-analysis method and the use of ballpark checks are techniques that will help you solve problems of many kinds, not just unit conversions.

Conversion between concentration units is so easy once you understand the above conversion principles, the units of concentration most frequently encountered in analytical chemistry are molarity, weight percent, volume percent, weight-to-volume percent, parts per million, and parts per billion. By recognizing the general definition of concentration given, it is easy to convert between concentration units. Table 1.3 shows some prefixes most commonly used in conversion between units. Table 1.4 shows relations between most commonly volume units.

Table 1.3: Some Prefixes for Multiples of SI Units

Factor	Prefix	Symbol	Example
1000,000,000 = 10^9	giga	G	1 gigameter (Gm) = 10^9 m
1000,000 = 10^6	mega	M	1 megameter (Mm) = 10^6 m
1000 = 10^3	kilo	k	1 kilogram (kg) = 10^3 g
100 = 10^2	hecto	h	1 hectogram (hg) = 100 g
10 = 10^1	deka	da	1 dekagram (dag) = 10 g
0.1 = 10^{-1}	deci	d	1 decimeter (dm) = 0.1 m
0.01 = 10^{-2}	centi	c	1 centimeter (cm) = 0.01 m
0.001 = 10^{-3}	milli	m	1 milligram (mg) = 0.001 g
0.000 001 = 10^{-6}	micro	μ	1 micrometer (mm) = 10^{-6} m
0.000 000 00 = 10^{-9}	nano	n	1 nanosecond (ns) = 10^{-9} s
0.000 000 000 001 = 10^{-12}	pico	p	1 picosecond (ps) = 10^{-12} s
0.000 000 000 000 001 = 10^{-15}	femto	f	1 femtosecond (fs) = 10^{-15} s

Table 1.4: Conversion between most commonly used volume units.

Unit	Symbol	Relation	Remark
Liter	L	1L = 1000 mL	
deciLiter	dL	1 dL = 100 mL 1 L = 10 dL	deci = 10^{-1} deciLiter = 10^{-1} L
milliLiter	mL	1 L = 1000 mL 1 dL = 100 mL	milli = 10^{-3} milliLiter = 10^{-3} L
Cubic meter	m ³	1 m ³ = 1000 L 1 m ³ = 10,000 dL	
Cubic decimeter	dm ³	1 m ³ = 1000 dm ³ 1 m ³ = 1000 L 1 dm ³ = 1 L	deci = 10^{-1} (deci...) ³ = 10^{-3} Cubic decimeter = 10^{-3} m ³
Cubic Centimeter	cm ³	1 m ³ = 1000 dm ³ 1 dm ³ = 1000 cm ³ 1 L = 1 dm ³ 1 L = 1000 mL 1 mL = 1 cm ³	centi = 10^{-2} (centi...) ³ = 10^{-6} Cubic centimeter = 10^{-6} m ³

Example 1.8

A concentrated solution of aqueous ammonia is 28.0% w/w NH₃ and has a density of 0.899 g/mL. What is the molar concentration of NH₃ in this solution?

SOLUTION:

We will use the general conversion equation starting from information we have, 28 % w/w means for every 100 g of solution it contain 28 g ammonia NH_3 , density of solution 0.899 g/mL and molecular weight of ammonia NH_3 is 17.03 g/mol. To convert to molarity we should reach the molarity expression moles per Liter:

$$\text{Molarity} = \frac{\text{Moles of solute}}{\text{Volume of solution (L)}}$$

To reach this expression we will put all possible conversion factors we will use, note the table shows two types for each conversion factor (factor 1) and (factor 2) we both are same but you have to justify it in the way that units cancel each other until you reach units expression you want (mol/L).

Table 1.5: All possible conversion factors we expect to use. Use factor 1 or factor 2 from each type:

Factor Description	Factor 1	Factor 2
Solution Density (g/mL)	$\frac{0.899 \text{ g Solution}}{1 \text{ mL Solution}}$	$\frac{1 \text{ mL Solution}}{0.899 \text{ g Solution}}$
Molecular Weight (g/mol)	$\frac{1 \text{ mol NH}_3}{17.03 \text{ g NH}_3}$	$\frac{17.03 \text{ g NH}_3}{1 \text{ mol NH}_3}$
Volume Conversion (mL/L)	$\frac{1000 \text{ mL Solution}}{1 \text{ L Solution}}$	$\frac{1 \text{ L Solution}}{1000 \text{ mL Solution}}$

If we arrange above conversion factors the following expression will reach:

$$\frac{28 \text{ g NH}_3}{100 \text{ g of solution}} \times \frac{0.899 \text{ g Solution}}{1 \text{ mL Solution}} \times \frac{1 \text{ mol NH}_3}{17.03 \text{ g NH}_3} \times \frac{1000 \text{ mL Solution}}{1 \text{ L Solution}} = 14.78 \text{ (mol/L) M NH}_3$$

The first part of the left side is the concentration that we have and the rest three are conversion factors.

There are many ways you can use to solve this kind of problem and we consider easiest and fastest one. To solve this problem using general molarity equations you have to assume you have 1 L of ammonia solution and continue you calculations based on this assumption and you will get the same result.

Example 1.9

The maximum allowed concentration of chloride in a municipal drinking water supply is 102 ppm Cl^- . When the supply of water exceeds this limit, it often has a distinctive salty taste. What is this concentration in moles Cl^- /liter?

SOLUTION

Using the same strategy start from information we have:

$$\frac{102 \cancel{\text{mg Cl}^-}}{1 \text{ L Solution}} \times \frac{1 \cancel{\text{g Cl}^-}}{1000 \cancel{\text{mg Cl}^-}} \times \frac{1 \text{ mol Cl}^-}{35.45 \cancel{\text{g Cl}^-}} = 0.002877 \text{ (mol/L) M Cl}^-$$

$$0.002877 \text{ M Cl}^- \times \frac{1000 \text{ mM Cl}^-}{1 \text{ M Cl}^-} = 2.877 \text{ mM Cl}^-$$

In all of incoming chapters instead of use the word “Solute” we will use new term “Analyte” because it will be more representative in scientific lab routine work. Analyte is defined as target substance in solution or the part side of solute we are concern in. If a solution of Sodium Chloride NaCl with a concentration of 0.1 M then if we analyze for Sodium only the say the analyte (Sodium - Na) concentration is 0.1 M Na.

1.7 Dilution of Concentrated Solutions

Dilution is one of the main preparation processes which used daily in all laboratories; any chemist should be familiar with dilution calculations to prepare a correct diluted solution.

For convenience, chemicals are sometimes bought and stored as concentrated solutions that must be diluted before use. Aqueous hydrochloric acid, for example, is sold commercially as a 12.0 M solution, yet it is most commonly used in the laboratory after dilution with water to a final concentration of 6.0 M or 1.0 M.



The key fact to remember when diluting a concentrated solution is that the number of moles of solute is constant; only the volume is changed by adding more solvent. Because the number of moles of solute can be calculated by multiplying molarity times volume, we can set up the following equation:

Moles of solute (constant) = Molarity \times Volume

$$= M_i \times V_i = M_f \times V_f$$

where M_i is the initial molarity, V_i is the initial volume, M_f is the final molarity, and V_f is the final volume after dilution. Rearranging this equation into a more useful form shows that the molar concentration after dilution (M_f) can be found by multiplying the initial concentration (M_i) by the ratio of initial and final volumes (V_i/V_f):

$$M_f = M_i \times \frac{V_i}{V_f}$$

Part (V_f/V_i) called dilution factor:

$$M_f = \frac{M_i}{DF} \iff M_i = M_f \times DF$$

$$\text{Where DF (Dilution Factor)} = \frac{V_f}{V_i} = \frac{M_i}{M_f}$$

Suppose, for example, that we dilute 50.0 mL of a solution of 2.00 M to a volume of 200.0 mL. The solution volume increases by a factor of four (from 50 mL to 200 mL), so the concentration of the solution must decrease by a factor of four (from 2.00 M to 0.500 M):

$$M_f = 2.00 \text{ M} \times \frac{50.0 \text{ mL}}{200.0 \text{ mL}} = 0.500 \text{ M}$$

$$\text{Dilution Factor DF} = \frac{V_f}{V_i} = \frac{200.0 \text{ mL}}{50.0 \text{ mL}} = 4$$

Dilution factor of value 4 means the solution concentration has been diluted by factor of 4, i.e the original solution concentration is equal four times the new concentration of the diluted solution

$$M_f = \frac{M_i}{DF} = \frac{2.00 \text{ M}}{4} = 0.500 \text{ M}$$

Example 1.10

How would you prepare 500.0 mL of 0.2500 M NaOH solution starting from a concentration of 1.000 M?

SOLUTION

The problem gives initial and final concentrations (M_i and M_f) and final volume (V_f) and asks for the initial volume (V_i) that we need to dilute. Rewriting the equation as gives the answer.

$$M_f = M_i \times \frac{V_i}{V_f} \iff V_i = \frac{V_f \times M_f}{M_i}$$

$$V_i = \frac{V_f \times M_f}{M_i} = \frac{500.0 \text{ mL} \times 0.2500 \text{ M}}{1.000 \text{ M}} = 125 \text{ mL}$$

This mean to prepare solution with concentration of 0.2500 M NaOH you have to transfer 125 mL from initial solution (1.000 M) and complete with solvent to 500.0 mL.

Note about the dilution roles:

- The volume units for both final volume (V_f) and initial volume (V_i) should be same, i.e if V_i is in (mL) unit then V_f is also in (mL) unit. If V_i is in Liter unit, also V_f should also have Liter unit...etc.

- The concentration (M_i) and (M_f) are not limited to molarity only, it could be generalized for any concentration unit, but under one condition they have to be same unit. The following equation used as general:

$$M_f = M_i \times \frac{V_i}{V_f} \iff C_f = C_i \times \frac{V_i}{V_f}$$

Where C_i is initial concentration, C_f is final concentration.

More Examples

Example 1.11

How many grams of Sodium Persulfate ($\text{Na}_2\text{S}_2\text{O}_8$) required to prepare a 1 L solution of Sodium Persulfate with concentration of 10% (w/v). This solution is widely used as oxidizing reagent for Total Organic Carbon analyzer (TOC).

SOLUTION 1 By Equations:

$$\%(\text{w/v}) = \frac{\text{g of solute}}{\text{mL of Solution}} \times 100\% \longrightarrow \text{g of solute} = \frac{\%(\text{w/v}) \times \text{mL of Solution}}{100\%}$$

$$\text{g of Na}_2\text{S}_2\text{O}_8 = \frac{10\%(\text{w/v}) \times 1000 \text{ mL of Solution}}{100\%} = 100 \text{ g of Na}_2\text{S}_2\text{O}_8$$

SOLUTION 2 By Conversions:

$$1 \text{ L} = 1000 \text{ mL}$$

$$1000 \text{ mL Solution} \times \frac{10 \text{ g of Na}_2\text{S}_2\text{O}_8}{100 \text{ mL of Solution}} = 100 \text{ g of Na}_2\text{S}_2\text{O}_8$$

Reagent is prepared by dissolving 100 g of Sodium Persulfate in solvent and complete to 1 L with solvent.

Example 1.12

What is the concentration of Hydrochloric Acid (HCl) bottle in Molarity unit (M), if the following information written on the bottle:

$$\text{M.Wt} = 36.46 \text{ g/mol}, d = 1.18 \text{ g/cm}^3, 37\% (\text{w/w})$$

SOLUTION

Molarity unit (M) is equal to mol/L by using conversion factor starting from information we have:

$$37\% (\text{w/w}) = \frac{37 \text{ g of HCl}}{100 \text{ g of Solution}} \longrightarrow$$

$$\frac{37 \text{ g of HCl}}{100 \text{ g of Solution}} \times \frac{1.18 \text{ g Solution}}{1 \text{ mL solution}} \times \frac{1000 \text{ mL Solution}}{1 \text{ L Solution}} \times \frac{1 \text{ mol HCl}}{36.46 \text{ g HCl}} = 11.97 \text{ g/mol (M) HCl}$$

Example 1.13

How to prepare a 500 mL solution of 2.0 N Sulfuric acid (H₂SO₄) from concentrated bottle of Sulfuric acid. If the following information written on Sulfuric acid bottle:

M.Wt = 98.08 g/mol, d= 1.84 g/cm³, 97% (w/w).

SOLUTION

In this example we have to dilute from concentrated bottle of Sulfuric acid (97% (w/w)) to lower concentration of 2.0 N.

1- The first step we have to find out the concentrated bottle concentration in unit Molarity.

2- Convert concentration from Molarity to Normality

3- Use dilution role to calculate the volume required from the concentrated bottle

To find the concentration of the concentrated bottle we start from information we have:

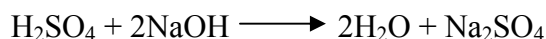
$$\text{Density} = 1.84 \text{ g/cm}^3 = 1.84 \text{ g/mL}$$

$$97\% \text{ (w/w)} = \frac{97 \text{ g of H}_2\text{SO}_4}{100 \text{ g of Solution}} \longrightarrow$$

$$\frac{97 \text{ g of H}_2\text{SO}_4}{100 \text{ g of Solution}} \times \frac{1.84 \text{ g of Solution}}{1 \text{ mL of Solution}} \times \frac{1000 \text{ mL Solution}}{1 \text{ L Solution}} \times \frac{1 \text{ mol H}_2\text{SO}_4}{98.08 \text{ g of H}_2\text{SO}_4} = 18.197 \text{ (mol/L) M of H}_2\text{SO}_4$$

The concentration of H₂SO₄ is 18.197 M

To convert this unit to Normality we have to find out the number of equivalents for H₂SO₄, Sulfuric acid is strong acid and always donate two hydrogen ions in all reactions for example:



In this case we say the number of equivalents for Sulfuric acid equals two (n=2). By substitute in Molarity to Normality equation:

$$N = n \times M$$

$$N = 2 \times 18.197 = 36.394 \text{ N of H}_2\text{SO}_4$$

Now we found the concentration of the Sulfuric acid and have to make dilutions by using dilution role:

Initial Concentration (C_i) = 36.394 N

Final Concentration (C_f) = 2.0 N

Final Volume (V_f) = 500 mL

Initial Volume (V_i) = ?

$$C_f = C_i \times \frac{V_i}{V_f} \longrightarrow V_i = \frac{C_f \times V_f}{C_i} = \frac{2.0 \times 500 \text{ mL}}{36.394 \text{ N}} = 27.5 \text{ mL}$$

This mean to prepare a solution of 2.0 N you have to transfer exactly 27.5 mL from concentrated bottle and dilute with solvent to total volume 500.0 mL.

Example 1.14

If we need to prepare a solution of NaOH with concentration of 20% (w/w) with total weight of solution equals to 2.0 Kg, how many grams of Sodium Hydroxide required.

SOLUTION

By using conversion factors starting from information we have:

$$20\%(w/w) = \frac{20 \text{ g of NaOH}}{100 \text{ g of Solution}}$$

$$1 \text{ Kg of solution} = 1000 \text{ g of solution} \longrightarrow$$

$$2.0 \text{ Kg of Solution} \times \frac{20 \text{ g of NaOH}}{100 \text{ g of Solution}} \times \frac{1000 \text{ g of Solution}}{1 \text{ Kg of Solution}} = 400.0 \text{ g of NaOH}$$

To prepare solution by dissolving 400.0 g of NaOH in suitable amount of solvent and dilute to 2.0 Kg.

Note:

To prepare solution in lab in practical work the process done by taking initial weight of the container need to prepare the solution in then transfer some solvent and 400.0 g of NaOH, shake and mix until all dissolved, then add solvent and complete to final weight on balance where:

$$\text{Final weight} = \text{Initial weight} + \text{Total Solution Volume (2.0 Kg)}$$

Example 1.15

A solution has been prepared by transfer 60 mL from Ortho-phosphoric acid 85 % (v/v) H_3PO_4 and dilute to 1.0 L, what is the concentration of the new solution.

SOLUTION

By using dilution role:

$$1.0 \text{ L} = 1000 \text{ mL}$$

$$C_f = C_i \times \frac{V_i}{V_f} = 85 \% (v/v) \times \frac{60.0}{1000 \text{ mL}} = 5.10 \% (v/v)$$

Note: This solution is generally used as a second oxidizing agent in Total Organic Carbon analyzer (TOC).

Example 1.16

A Total Petroleum Hydrocarbon (TPH) standard solution is prepared by dissolving amount of mineral oil (motor oil) in organic solvent like n-hexane or chloroform. Explain how to prepare TPH standard in 100 mL volume with concentration of 50,000 ppm (w/v).

SOLUTION

By using conversion factor starting from information we have:

$$50,000 \text{ ppm} = \frac{50000 \text{ mg of TPH}}{1 \text{ L of Solution}}$$

$$100 \text{ mL} = 0.100 \text{ L}$$

$$\frac{50000 \text{ mg of TPH}}{1 \text{ L of Solution}} \times \frac{1 \text{ g TPH}}{1000 \text{ mg TPH}} \times \frac{1 \text{ L of Solution}}{1000 \text{ mL of Solution}} \times 100 \text{ mL of Solution} = 5.0 \text{ g}$$

To prepare TPH standard weigh exactly 5.0 g of mineral oil and complete to total volume 100 mL.

Example 1.17

How many moles Sodium Sulfate Na_2SO_4 exists in a 250 mL solution have concentration 15% (w/v).

SOLUTION

Molecular Weight of $\text{Na}_2\text{SO}_4 = (22.99 \times 2) + 32.07 + (16.0 \times 4) = 142.04 \text{ g/mol}$

$$250 \text{ mL of Solution} \times \frac{15 \text{ g of Na}_2\text{SO}_4}{100 \text{ mL of Solution}} \times \frac{1 \text{ mole of Na}_2\text{SO}_4}{142.04 \text{ g Na}_2\text{SO}_4} = 0.264 \text{ mol of Na}_2\text{SO}_4$$

Example 1.18

How to prepare 200 mL of solution with concentration of 1% (w/v) NaOH starting from NaOH 20 % (w/v).

SOLUTION

In this example you have a simple dilution process, use dilution equation to calculate the amount required:

$$C_f \times V_f = C_i \times V_i$$

Where in this case:

$$C_f = 1 \% \text{ (w/v) NaOH}$$

$$C_i = 20\% \text{ (w/v) NaOH}$$

$$V_f = 250 \text{ mL}$$

V_i = Volume required from 20% NaOH solution

Substitute above values in equation:

$$V_i = \frac{C_f}{C_i} \times V_f = \frac{1 \% \text{ (w/v)}}{20 \% \text{ (w/v)}} \times 200 \text{ mL} = 10 \text{ mL}$$

Volume of 10 mL required, to prepare the solution transfer exactly 10 mL from 20% (w/v) NaOH solution to 200 mL volumetric flask and dilute with solvent to the mark.

Example 1.19

How to prepare 500 g solution of 5 % (w/w) NaOH starting from 50% (w/w) NaOH solution.

SOLUTION

In this example dilution equation will be used but instead of put volume we will put weight

$$C_f \times W_f = C_i \times W_i$$

Where in this case:

$$C_f = 5 \% \text{ (w/w) NaOH}$$

$$C_i = 50\% \text{ (w/w) NaOH}$$

$$W_f = 500 \text{ g of } 5 \% \text{ (w/w) solution}$$

W_i = Volume required from 50% (w/w) NaOH solution

Substitute above values in equation:

$$W_i = \frac{C_f}{C_i} \times W_f = \frac{5 \% \text{ (w/v)}}{50 \% \text{ (w/v)}} \times 500 \text{ g} = 50 \text{ g}$$

Weight of 50 g from 50%(w/w) NaOH required to prepare solution of 5% (w/w) NaOH.

Problems

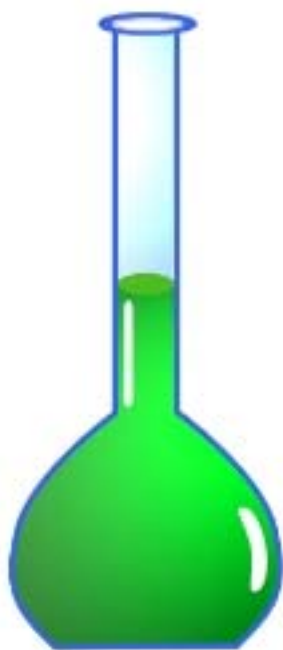
To make sure you understand basic principles for solution preparation always try to solve many problems as much as you can, try to solve the following problems:

Hint: To help you get accurate result use **Chemiasoft-Calculator** and **Chemiasoft-Molecular Weight Calculator** software which will help you to make your calculations easy, you can download it from internet web site <http://www.chemiasoft.com>, those softwares are freeware under GPL and easy to use.

- 1-1)** Calculate the concentration of solute in each of the following solutions, express concentration in Molarity (M):
 - a. Sodium chloride solution prepared by dissolves 25 g of Sodium Chloride NaCl in 250 mL distilled water.
 - b. Sodium Persulfate $\text{Na}_2\text{S}_2\text{O}_8$ prepared by dissolve 100 g of $\text{Na}_2\text{S}_2\text{O}_8$ in 250 mL of water.
 - c. Sodium hydroxide solution 25 % (w/v) NaOH
 - d. $\text{Na}_2\text{S}_2\text{O}_3$ 1000 ppm (mg/L)
- 1-2)** Blank solution for metals analysis on ICP-OES is normally 2% Nitric acid (HNO_3) solution, how to prepare 1 liter of the blank solution from concentrated bottle of Nitric acid if the following information appear on the concentrated bottle. 69% (w/w), $d=1.408 \text{ g/cm}^3$, M.W = 63.01288 g/mol.
- 1-3)** Diesel range organic (DRO) test in environmental laboratories done using Gas Chromatography (GC), standard prepared from commercial diesel liquid available from any fuel station, describe how to prepare 100 mL of 5,000 ppm (mg/L) solution from diesel using n-hexane as solvent.
- 1-4)** What is the normality of the following:
 - a. 0.12 M H_2SO_4
 - b. 2.35 M H_3PO_4
 - c. 0.5641 g of acid (Equivalent Weight = 187.3) in 250 mL of solution
 - d. 1.789 g of Sodium carbonate (Na_2CO_3) in 500 mL solution
- 1-5)** How many grams of MgF_2 are needed to prepare 250 mL of 1.50 M MgF_2
- 1-6)** 37.5 g of NaOH is dissolved in enough water to create 500. mL of solution. What is the molarity of the solution.
- 1-7)** Express the following solution concentrations in % (w/v)

- a. 5 N of NaOH solution
 - b. 2000 ppm of NaCl
 - c. 3 M of Na_2CO_3
- 1-8)** The density of concentrated ammonia, which is 28.0 % w/w NH_3 , is 0.899 g/mL. What volume of this reagent should be diluted to 1,000 mL to make a solution that is 0.036 M in NH_3 ?
- 1-9)** How many grams of perchloric acid, HClO_4 , are contained in 37.6 g of 70.5 wt % aqueous perchloric acid? How many grams of water are in the same solution?
- 1-10)** The concentration of sugar (glucose, $\text{C}_6\text{H}_{12}\text{O}_6$) in human blood ranges from about 80 mg/100 mL before meals up to 120 mg/100 mL after eating. Find the molarity of glucose in blood before and after eating.
- 1-11)** An aqueous solution of antifreeze contains 6.067 M ethylene glycol ($\text{HOCH}_2\text{CH}_2\text{OH}$, FM 62.07) and has a density of 1.046 g/mL.
- a. Find the mass of 1.000 L of this solution and the number of grams of ethylene glycol per liter.
 - b. Find the molality of ethylene glycol in this solution.
- 1-12)** How many grams of boric acid ($\text{B}(\text{OH})_3$, FM 61.83) should be used to make 2.00 L of 0.05 M solution? What kind of flask is used to prepare this solution?
- 1-13)** Describe how you would prepare approximately 2 L of 0.050 0 m boric acid, $\text{B}(\text{OH})_3$.
- 1-14)** What is the maximum volume of 0.25 M sodium hypochlorite solution (NaOCl , laundry bleach) that can be prepared by dilution of 1.00 L of 0.80 M NaOCl ?
- 1-15)** How many grams of 50 wt % NaOH (FM 40.00) should be diluted to 1.00 L to make 0.10 M NaOH? (Answer with 2 digits.)
- 1-16)** A bottle of concentrated aqueous sulfuric acid, labeled 98.0 wt % H_2SO_4 , has a concentration of 18.0 M.
- a. How many milliliters of reagent should be diluted to 1.000 L to give 1.00 M H_2SO_4 ?
 - b. Calculate the density of 98.0 wt % H_2SO_4 .

Chapter 2: Types of Stock Standard Solutions



2.1 Introduction

In chapter one all kinds of concentration units have been explained with many examples, we have tried through many exercises which should help you enough to understand the basics of solution preparation.

In this chapter we will go through different kinds of standard solutions and we will consider many examples as in chapter one.

There are three major kinds of stock standard solutions (depend on chemical formula) **Ionic**, **Element** and **Formula** standards. Sections 2.2, 2.3 and 2.4 explain each kind of those standards. Some examples included for explaining the calculations.



2.2 Ionic Standard Solutions:

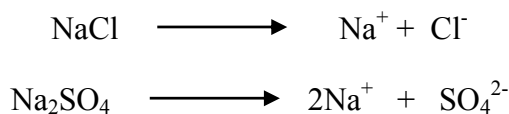
2.2.1 Ionic Compounds:

Ionic compounds are basically defined as being compounds where two or more ions are held next to each other by electrical attraction. One of the ions has a positive charge (called a "cation") and the other has a negative charge ("anion"). Cations are usually metal atoms and anions are either nonmetals or polyatomic ions (ions with more than one atom).

Ions can be single atoms, as the sodium and chloride in common table salt sodium chloride, or more complex groups such as the carbonate in calcium carbonate. But to be considered an ion, they must carry a positive or negative charge. Thus, in an ionic bond, one 'bonder' must have a positive charge and the other a negative one. By sticking to each other, they resolve, or partially resolve, their separate charge imbalances. Positive to positive and negative to negative ionic bonds do not occur.

Ionic standard solutions are mostly used in analysis of all kind of ions, anions such as chloride (Cl^-), bromide (Br^-), sulfate (SO_4^{2-}) or cations such as sodium (Na^+), magnesium (Mg^{2+}) and potassium (K^+). The analysis procedure is either in wet-chemistry by titration or by using instrument like ion-chromatography or spectrophotometer or else. But regardless of the procedure used in the analysis the accuracy in the analysis depends on the accuracy of standards.

To prepare stock standard solution of ion analyte, first we have to find a salt starting reagent either in liquid or solid state contain such ion then make sure the salt is fully dissociated in water such as sodium chloride (NaCl) and sodium sulfate (Na₂SO₄).



Sodium chloride dissociate in water into one sodium cation (Na⁺) and one chloride anion (Cl⁻) as per equation above. Sodium sulfate dissociate into two sodium cations and one sulfate anion.

After find the starting reagent to prepare standard solution check the chemical formula of the substance and find how many ion(s) are produced from dissociation of single molecule. Example each molecule of NaCl produces one sodium ion (Na⁺) but each molecule of Na₂SO₄ produce two sodium ions. Use the conversion factor equation to make calculations.

We consider some examples below will show how to prepare and make calculations for different kinds of ionic standard solutions.

Example 2.1

Calculate amount required to prepare 500 mL solution of 1000 ppm (mg/L) Na starting from sodium chloride (NaCl).

SOLUTION

We will use conversion factor and we will start with conversion we will prepare:

Molecular weight for NaCl = 58.44 g/mol

Atomic weight for Na = 22.99 g/mol

Each 58.44 g of NaCl contain 22.99 g of Na by convert grams to milligrams

Each 58.44 mg of NaCl contain 22.99 mg of Na

$$\frac{1000 \text{ mg Na}}{\text{L}} \times \frac{1 \text{ g Na}}{1,000 \text{ mg Na}} \times \frac{58.44 \text{ g NaCl}}{22.99 \text{ g Na}} \times 0.5 \text{ L} = 1.271 \text{ g NaCl}$$

Good Practice: Calculate amount required to prepare 1000 mL of 2000 ppm (mg/L) Na starting from Na₂SO₄.

Example 2.2

Calculate how to prepare sulfate (SO₄²⁻) stock standard solution 1000 ppm (mg/L) in 1 Liter starting from magnesium sulfate hexahydrate (MgSO₄·7H₂O).

SOLUTION

First will calculate the molecular weight for starting reagent formula and for sulfate ion

Molecular weight for $\text{MgSO}_4 \cdot 7\text{H}_2\text{O} = 246.48 \text{ g/mol}$

Formula weight for sulfate ion (SO_4^{2-}) = 96.06 g/mol

From starting reagent formula $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ each molecule produce after dissociate in water one sulfate anion (SO_4^{2-}) i.e each 246.48 grams of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ have 96.06 grams of sulfate anion. Use this relation to write the conversion factor. Start always from the concentration we need to prepare 1000 ppm (mg/L).

$$\frac{1,000 \text{ mg SO}_4^{2-}}{\text{L}} \times \frac{1 \text{ g SO}_4^{2-}}{1,000 \text{ mg SO}_4^{2-}} \times \frac{246.48 \text{ g MgSO}_4 \cdot 7\text{H}_2\text{O}}{96.06 \text{ g SO}_4^{2-}} \times 1 \text{ L} = 2,565.90 \text{ mg} = 2.5659 \text{ g MgSO}_4 \cdot 7\text{H}_2\text{O}$$

To prepare stock solution dissolve 2.5659 grams of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in small amount of solvent then complete to total volume 1 Liter.

Example 2.3

Nitrate (NO_3^-) anion solution prepared by dissolving 3.0 g of KNO_3 in 250 mL of water. What is the concentration of Nitrate ion, express the concentration in Molarity and ppm.

SOLUTION

Molecular weight of $\text{KNO}_3 = 101.10 \text{ g/mol}$

Formula weight of $\text{NO}_3^- = 62.00 \text{ g/mol}$

Starting from the information we have:

$$\frac{3.0 \text{ g KNO}_3}{0.250 \text{ L}} \times \frac{62.00 \text{ g NO}_3^-}{101.10 \text{ g KNO}_3} \times \frac{1000 \text{ mg NO}_3^-}{1.0 \text{ g NO}_3^-} = 7359.05 \text{ mg/L NO}_3^-$$

$$\frac{3.0 \text{ g KNO}_3}{0.250 \text{ L}} \times \frac{1 \text{ mol KNO}_3}{101.10 \text{ g KNO}_3} \times \frac{1 \text{ mol NO}_3^-}{1 \text{ mol KNO}_3} = 0.1187 \text{ mol/L (M) NO}_3^-$$

Example 2.4

A student needs to prepare mixed anion standard solution 1 Liter to be used in Ion Chromatography contains the following:

- 1- Sulfate (SO_4^{2-}) 10,000 ppm
- 2- Nitrate (NO_3^-) 1,000 ppm
- 3- Bromide (Br^-) 1,000 ppm

Student has the following reagent in his lab, Aluminum sulfate hexadecahydrate $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$, Sodium bromide NaBr , Calcium nitrate $\text{Ca}(\text{NO}_3)_2$.

Explain to the student how to prepare stock standard.

SOLUTION

First we have to find a source for each ion as per the following table:

Ion	Source	Concentration (ppm)	Number of ion in each molecule
Sulfate (SO_4^{2-})	Aluminum sulfate hexadecahydrate $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$	10,000	3
Nitrate (NO_3^-)	Calcium nitrate $\text{Ca}(\text{NO}_3)_2$	1,000	2
Bromide (Br^-)	Sodium bromide NaBr	1,000	1

Above table shows each ion in stock standard and its starting reagent source and concentration in ppm (mg/L). The last column shows number of ions produced from each molecule of starting reagents after dissociation in water.

To calculate the amount required we assume all starting reagents are pure 100 % w/w. Start calculate from the target concentration:

a) Sulfate (SO_4^{2-})

Molecular weight of $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O} = 630.40 \text{ g/mol}$

$$\frac{10,000 \text{ mg SO}_4^{2-}}{\text{L}} \times \frac{1 \text{ g SO}_4^{2-}}{1,000 \text{ mg SO}_4^{2-}} \times \frac{630.40 \text{ g Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}}{3 \times 96.06 \text{ g SO}_4^{2-}} \times 1 \text{ L} = 21.8752 \text{ g Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$$

b) Nitrate (NO_3^-)

Molecular weight of $\text{Ca}(\text{NO}_3)_2 = 164.09 \text{ g/mol}$

$$\frac{1,000 \text{ mg NO}_3^-}{\text{L}} \times \frac{1 \text{ g NO}_3^-}{1,000 \text{ mg NO}_3^-} \times \frac{164.09 \text{ mg Ca}(\text{NO}_3)_2}{2 \times 62.00 \text{ mg NO}_3^-} \times 1 \text{ L} = 1.3233 \text{ g Ca}(\text{NO}_3)_2$$

c) Bromide (Br^-)

Molecular weight of $\text{NaBr} = 102.89 \text{ g/mol}$

$$\frac{1,000 \text{ mg Br}^-}{\text{L}} \times \frac{1 \text{ g Br}^-}{1,000 \text{ mg Br}^-} \times \frac{102.89 \text{ g NaBr}}{79.9000 \text{ g Br}^-} \times 1 \text{ L} = 1.2877 \text{ g NaBr}$$

To prepare standard we weigh exactly 21.8752 g, 1.3233 g and 1.2877 g from $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$, $\text{Ca}(\text{NO}_3)_2$ and NaBr respectively and transfer all to same 1 liter volumetric flask then add solvent (water) to dissolve all substances then we complete to marked volume with solvent.

Example 2.5

A solution prepared by add 5.0 g of KNO_3 and 5.0 g of $\text{Hg}(\text{NO}_3)_2$ in 1 L volumetric flask and complete with water to volume, what is the concentration of Nitrate anion (NO_3^-) in the final solution? Express concentration in Molarity unit.

SOLUTION

First we calculate how many grams or how many moles of nitrate ion in the solution from each source reagent, we start always from the information we know:

Molecular weight of $\text{KNO}_3 = 101.10 \text{ g/mol}$

$$5.0 \text{ g KNO}_3 \times \frac{1 \text{ mol KNO}_3}{101.10 \text{ g KNO}_3} \times \frac{1 \text{ mol NO}_3^-}{1 \text{ mol KNO}_3} = 0.04946 \text{ moles} = 49.46 \text{ mmoles of NO}_3^-$$

Molecular weight of $\text{Hg}(\text{NO}_3)_2 = 324.60 \text{ g/mol}$

$$5.0 \text{ g Hg}(\text{NO}_3)_2 \times \frac{1 \text{ mol Hg}(\text{NO}_3)_2}{324.60 \text{ g Hg}(\text{NO}_3)_2} \times \frac{2 \text{ mol NO}_3^-}{1 \text{ mol Hg}(\text{NO}_3)_2} = 0.03080 \text{ moles} = 30.80 \text{ mmoles of NO}_3^-$$

Total number of moles of nitrate ions from both source =

$$49.46 \text{ mmoles} + 30.80 \text{ mmoles} = 80.26 \text{ mmoles} = 0.08026 \text{ moles NO}_3^-$$

$$\text{Molarity} = \frac{\text{Moles}}{\text{Solution Volume (L)}} = \frac{0.08026 \text{ moles NO}_3^-}{1 \text{ L}} = 0.08026 \text{ M NO}_3^-$$

Example 2.6

Sulfide (S^{2-}) ion solution prepared from sodium sulfide Na_2S solid with purity of 30% (w/w). How many grams of sodium sulfide required to prepare 500 mL of 2,000 ppm sulfide (S^{2-}) stock standard solution.

SOLUTION

Purity of starting reagent is important, 30% w/w means for each 100 grams of sodium sulfide powder there are 30 grams of sodium sulfide, we will start calculations from the target concentration 2,000 ppm:

$$\begin{aligned} \frac{2,000 \text{ mg S}^{2-}}{\text{L}} \times \frac{1 \text{ g S}^{2-}}{1,000 \text{ mg S}^{2-}} \times \frac{1 \text{ mol S}^{2-}}{32.07 \text{ g S}^{2-}} \times \frac{1 \text{ mol Na}_2\text{S}}{1 \text{ mol S}^{2-}} \times \frac{78.05 \text{ g Na}_2\text{S}}{1 \text{ mol Na}_2\text{S}} \times \frac{100 \text{ g Na}_2\text{S Powder}}{30 \text{ g Na}_2\text{S}} \times 0.500 \text{ L} = \\ = 8.1125 \text{ g of Na}_2\text{S powder} \end{aligned}$$

To prepare standard weigh exactly 8.1125 g of Na_2S powder dissolved it in water then complete to total volume 500 mL.

Example 2.7

A solution prepared by mixing 50 mL from 0.5 M orthophosphate (PO_4^{3-}) and 35 mL from 5,000 ppm (mg/L) PO_4^{3-} solutions then completed to 250 mL total volume. What is the final concentration of phosphate anion PO_4^{3-} in both ppm and molarity.

SOLUTION

First we calculate number of moles of orthophosphate ion we took from each solution:

- 50 mL of 0.5 M PO_4^{3-} solution (M = mol/L)

$$\frac{0.5 \text{ mol } \text{PO}_4^{3-}}{\text{L}} \times 0.050 \text{ L} = 0.025 \text{ mol } \text{PO}_4^{3-}$$

- 35 mL of 5,000 ppm PO_4^{3-} solution (ppm = mg/L)

$$\frac{5,000 \text{ mg } \text{PO}_4^{3-}}{\text{L}} \times \frac{1 \text{ g } \text{PO}_4^{3-}}{1,000 \text{ mg } \text{PO}_4^{3-}} \times \frac{1 \text{ mol } \text{PO}_4^{3-}}{94.97 \text{ g } \text{PO}_4^{3-}} \times 0.035 \text{ L} = 0.00184 \text{ mol } \text{PO}_4^{3-}$$

Total number of moles = $0.025 + 0.00184 = 0.02684 \text{ mol } \text{PO}_4^{3-}$

Calculate concentration of final solution in Molarity (M):

$$\text{Molarity} = \frac{\text{moles}}{\text{L}} = \frac{0.02684 \text{ mols } \text{PO}_4^{3-}}{0.250 \text{ L}} = 0.1074 \text{ M } \text{PO}_4^{3-}$$

Calculate concentration in ppm(mg/L):

$$0.02684 \text{ mol } \text{PO}_4^{3-} \times \frac{94.97 \text{ g } \text{PO}_4^{3-}}{1 \text{ mol } \text{PO}_4^{3-}} \times \frac{1,000 \text{ mg } \text{PO}_4^{3-}}{1 \text{ g } \text{PO}_4^{3-}} \times \frac{1}{0.250 \text{ L}} = 10,195.97 \text{ ppm (mg/L) } \text{PO}_4^{3-}$$

Final concentration of solution is 0.1074 M or 10,195.97 ppm PO_4^{3-}

Example 2.8

Nitrite anion (NO_2^-) stock solution prepared from Sodium nitrite NaNO_2 , the concentration of nitrite are expressed in terms of nitrogen element. For example if we said Nitrite-N 1,000 ppm mg N/L means 1 liter of the solution contains one thousand milligrams of nitrogen element sourced from nitrite anion.

Notice: Express nitrite and nitrate anions concentrations by nitrogen element source make it easy for comparison between them; most of national environmental protection agencies for water in many countries around the world are expressing thus kind of ions by nitrogen element. **Standard Methods for Examination of Water and Wastewater** book is also using this expression.

How many grams of Sodium nitrite NaNO_2 powder required to prepare 1 liter of Nitrite-N solution with concentration of 1,000 mg N/L if the purity of NaNO_2 powder is 97.1 % w/w.

SOLUTION

Calculation is depend on nitrogen element in Sodium nitrite, there is only on nitrogen atom produced from each molecule of Sodium nitrite

$$\frac{1,000 \text{ mg N}}{\text{L}} \times \frac{1 \text{ g N}}{1,000 \text{ mg N}} \times \frac{1 \text{ mol N}}{14.00 \text{ g N}} \times \frac{1 \text{ mol NO}_2^-}{1 \text{ mol N}} \times \frac{1 \text{ mol NaNO}_2}{1 \text{ mol NO}_2^-} \times \frac{69.00 \text{ g NaNO}_2}{1 \text{ mol NaNO}_2} \times \frac{100 \text{ g NaNO}_2 \text{ pwd}}{97.1 \text{ g NaNO}_2} =$$

$$= 5.0758 \text{ g of NaNO}_2 \text{ powder}$$

Good practice: resolve example 2.8 but change Sodium nitrite (NaNO_2) with Ammonium nitrite (NH_4NO_2).

2.3 Element Standard Solutions:

Element stock standard solutions used in the analysis of elements by analytical instruments like Inductively Coupled Plasma (ICP), Flame Photometer (FP) and Atomic Absorption (AA) which are the most commonly used in analysis of elements. Following are some examples for preparations of elements stock standard solutions.

Example 2.10

How many grams required from Iron (III) chloride $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ powder to prepare Iron (Fe) stock standard solution in 1 Liter with concentration of 1,000 ppm Fe if the purity of Iron (III) chloride is 96.5% w/w?

SOLUTION

Each molecule of Iron (III) chloride contains one mole of iron (Fe) element:

Molecular weight of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O} = 270.30 \text{ g/mol}$

Atomic weight of Fe = 55.85 g/mol

$$\frac{1,000 \text{ mg Fe}}{\text{L}} \times \frac{1 \text{ g Fe}}{1,000 \text{ mg Fe}} \times \frac{1 \text{ mol Fe}}{55.85 \text{ g Fe}} \times \frac{1 \text{ mol FeCl}_3 \cdot 6\text{H}_2\text{O}}{1 \text{ mol Fe}} \times$$

$$\frac{270.30 \text{ g FeCl}_3 \cdot 6\text{H}_2\text{O}}{1 \text{ mol FeCl}_3 \cdot 6\text{H}_2\text{O}} \times \frac{100 \text{ g FeCl}_3 \cdot 6\text{H}_2\text{O} \text{ pwd}}{96.5 \text{ g FeCl}_3 \cdot 6\text{H}_2\text{O}} = 5.0153 \text{ g of FeCl}_3 \cdot 6\text{H}_2\text{O} \text{ powder}$$

To prepare stock solution it required to weight 5.0153 g of Iron (III) chloride powder and dissolve it in small amount of water then complete to volume 1 L.

Example 2.11

A 500 mL of Mercury (Hg) solution prepared by mixing 2.45 g of mercury chloride (HgCl₂) and 4.68 g of mercury sulfate (HgSO₄) in one container then dissolve and complete to volume with solvent 2% nitric acid. What is the final concentration Mercury (Hg) in the final solution? Express concentration in Molarity and % (w/v).

SOLUTION

Each mole of either mercury chloride or mercury sulfate contain one mole of mercury element (Hg)

Molecular weight of mercury chloride (HgCl₂) = 271.50 g/mole

Molecular weight of mercury sulfate (HgSO₄) = 296.65 g/mole

Atomic weight of mercury element (Hg) = 200.59 g/mole

Calculate number of total number of moles of mercury element from each source

Mercury from mercury chloride (HgCl₂):

$$2.45 \text{ g HgCl}_2 \times \frac{1 \text{ mol HgCl}_2}{271.50 \text{ g HgCl}_2} \times \frac{1 \text{ mol Hg}}{1 \text{ mol HgCl}_2} = 0.00902 \text{ mole} = 9.02 \text{ mmol Hg}$$

Mercury from mercury sulfate (HgSO₄):

$$4.68 \text{ g HgSO}_4 \times \frac{1 \text{ mol HgSO}_4}{296.65 \text{ g HgSO}_4} \times \frac{1 \text{ mol Hg}}{1 \text{ mol HgSO}_4} = 0.0158 \text{ mole} = 15.8 \text{ mmol Hg}$$

Total number of millimoles = 9.02 + 15.8 = 24.8 mmol = 0.0248 moles mercury (Hg) in solution

Concentration of mercury in solution in molarity (M)

$$\text{Molarity} = \frac{\# \text{ of moles of Hg}}{\text{Solution volume (L)}} = \frac{0.0248 \text{ moles Hg}}{0.500 \text{ L}} = 0.0496 \text{ M Hg}$$

To concentration of mercury in solution in % (w/v) first we calculate grams of mercury in the solution:

Weight of mercury(Hg) = molecular weight × number of moles = 200.59 g/mol × 0.0248 moles = 4.97 g Hg

$$\%(\text{w/v}) = \frac{\text{g of solute}}{100 \text{ mL of Solution}} = \frac{\text{g of solute}}{\text{mL of Solution}} \times 100\% = \frac{4.97 \text{ g Hg}}{500 \text{ mL}} \times 100\% = 0.994\% (\text{w/v}) \text{ Hg}$$

Good Practice: Calculate the concentration of potassium and mercury in examples (2.5).

Example 2.13

A student has got three stock standard solutions of 3 different elements, zinc (Zn) 2000 ppm, cadmium (Cd) 1500 ppm and lead (Pb) 1000 ppm. A student took 10 mL from each solution and transfers it to 200 mL volumetric flask then completed to total volume with solvent. What is the final concentration of each element in the diluted mix solution?

SOLUTION

In this example simple dilution process was made, to calculate the concentration of each element in the final solution use dilution equation from chapter one:

$$C_f \times V_f = C_i \times V_i$$

Final concentration of zinc (Zn)

$$C_f = \frac{C_i \times V_i}{V_f} = \frac{2000 \text{ ppm} \times 10 \text{ mL}}{200 \text{ mL}} = 100 \text{ ppm Zn}$$

Final concentration of cadmium (Cd):

$$C_f = \frac{C_i \times V_i}{V_f} = \frac{1500 \text{ ppm} \times 10 \text{ mL}}{200 \text{ mL}} = 75 \text{ ppm Cd}$$

Final concentration of cadmium (Pb):

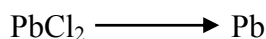
$$C_f = \frac{C_i \times V_i}{V_f} = \frac{1000 \text{ ppm} \times 10 \text{ mL}}{200 \text{ mL}} = 50 \text{ ppm Pb}$$

Example 2.14

Lead chloride (PbCl₂) powder has purity of 89% w/w, a chemist need to prepare 1 L of 0.5 M lead (Pb) stock solution. How many grams of PbCl₂ required for preparing such stock solution.

SOLUTION

Each mole of lead chloride contains one mole of lead (Pb) element:



Molarity of lead (Pb) = 0.5 M

Total volume (L) = 1 L

Purity of PbCl₂ = 89% (w/w) → each 100 grams of PbCl₂ powder contains 89 grams of PbCl₂

Starting from concentration required as first item in conversion factor equation:

$$\frac{0.5 \text{ mol Pb}}{\text{L}} \times \frac{1 \text{ mole PbCl}_2}{1 \text{ mole Pb}} \times \frac{278.1054 \text{ g PbCl}_2}{1 \text{ mole PbCl}_2} \times \frac{100 \text{ g PbCl}_2 \text{ powder}}{89 \text{ g PbCl}_2} = 156.24 \text{ g of PbCl}_2 \text{ powder}$$

It required 156.24 grams of lead chloride powder to prepare stock solution, dissolve it in solvent and complete to total volume 1 Liter.

2.3 Formula Standard Solutions:



Formula stock standard solutions means the analyte is the molecular formula of starting reagent. In ion standard solutions calculations are based on the analyte ion formula, also for elements stock solutions the calculations are based on the element atomic weight and number of elements in molecular formula. In this kind of standard the analyte is molecular formula of the starting reagent regardless if it dissociates in

solvent or not.

Examples of stock standard solutions:

Stock Standard Type	Example Solution
Ion	1000 ppm Sulfate (SO_4^{2-})
Element	1000 ppm Iron (Fe)
Formula	1000 ppb Naphthalene.

Following are some examples of formula standards.

Example 2.15

Naphthalene (C_{10}H_8) is one of aromatic hydrocarbons measured by GC-MS. If molecular weight of naphthalene is 128.6 g/mol; how many milligrams are required to prepare 100 mL of 2,000 ppb stock standard solution of naphthalene from powder Naphthalene (purity of 91.5 % w/w)?

SOLUTION

Concentration of naphthalene = 2,000 ppb ($\mu\text{g/L}$)

Purity of naphthalene = 91.5 % w/w \longrightarrow each 100 mg of Naphthalene powder contains 91.5 mg of pure naphthalene.

$$\frac{2,000 \mu\text{g Naphthalene}}{\text{L}} \times \frac{1 \text{ mg Naphthalene}}{1000 \mu\text{g Naphthalene}} \times \frac{100 \text{ mg Naphthalene powder}}{91.5 \text{ mg Naphthalene}} = 2.1858 \text{ mg naphthalene powder}$$

Example 2.15

Kerosene Range Organic (KRO) tested in all environmental and petroleum laboratories. The standard of KRO is prepared from commercial Kerosene fuel available from fuel station. How many grams required from kerosene to prepare 250 mL of 5000 ppm of KRO?

SOLUTION

KRO concentration = 5,000 ppm (mg/L)

Total volume = 250 mL = 0.250 L

$$\frac{5,000 \text{ mg KRO}}{\text{L}} \times \frac{1 \text{ g KRO}}{1,000 \text{ mg KRO}} \times 0.250 \text{ L} = 1.2500 \text{ g KRO}$$

Example 2.16

How many grams of methanol (CH₃OH) are contained in 0.100 L of 1.71 M aqueous methanol (i.e., 1.71 mol CH₃OH/L solution)?

SOLUTION

Molecular weight of methanol (CH₃OH) = 32.042 g/mol

$$\frac{1.71 \text{ mole}}{\text{L}} \times \frac{32.042 \text{ g methanol}}{1 \text{ mole methanol}} \times 0.100 \text{ L} = 5.4792 \text{ g methanol}$$

Practical & Challenge Problems

- 2-1)** A chemist trying to prepare Ammonia-N stock solution from ammonium chloride NH₄Cl with concentration of 100 mg-N /L as ammonia in 1 liter volumetric flask. Can you explain to the chemist how to make calculation for this solution?
- 2-2)** Antimony (Sb) is one of the most toxic element measured on Inductively Coupled Plasma (ICP), how many grams required to prepare stock solution of antimony (Sb) 1000 ppm in 1 liter from antimony potassium tartarate () which has purity of 96.3 % w/w?
- 2-3)** A chemist prepared 250 mL of 2,000 ppm stock solution of mercury (Hg) from mercury sulfate (HgSO₄) as a QC standard to be used in metals analysis by ICP, then he transfer accurately 0.5 mL of stock standard an diluted to 250 mL. Diluted solution measured on ICP compared with certified reference mercury standard and he found the concentration of final solution is 3,543 ppb (µg/L). What is the purity of mercury sulfate solid powder? how many grams required from mercury sulfate to prepare same stock standard of mercury with exact concentration 2,000 ppm?

- 2-4)** What is the concentration of chloride ion (Cl^-) in stock solution of silver (Ag) prepared from silver chloride (AgCl) with concentration of silver equals to 1,000 mg/L. Express concentration of chloride ion in molarity.
- 2-5)** Iron metal solution prepared by mixing two starting reagents; iron (III) chloride (FeCl_3) (purity 95.6% w/w) and iron (III) sulfate ($\text{Fe}_2(\text{SO}_4)_3$) (purity 88.5% w/w) in one liter of water, if the final concentration of chloride in the solution was 5,124 ppm Cl^- .
- How many grams of each starting reagents was used in the preparation?
 - What is the concentration of iron in the solution?
- 2-6)** An analyst wishes to add 256 mg of Cl^- to the reaction mixture. How many milliliter of 0.217 M BaCl_2 should be added?
- 2-7)** The concentration of lead in an industrial waste stream is 0.28 ppm. What is its molar concentration?
- 2-8)** A 250.0 mL aqueous solution contains 45.1 μg of a pesticide. Express the pesticide's concentration in weight percent, parts per million, and parts per billion.
- 2-9)** 5.0 grams of mineral oil are transferred to 100 mL volumetric flask and completed to volume with chloroform, the density of the final solution is 1.45 g/cm³, if the density of the chloroform is 1.5 g/mL; what is the density of the mineral oil?
- 2-10)** Calculate the molarity of a potassium dichromate solution prepared by placing 9.67 g of $\text{K}_2\text{Cr}_2\text{O}_7$ in a 100 mL volumetric flask, dissolving, and diluting to the calibration mark.
- 2-11)** For each of the following, explain how you would prepare 1.0 L of solution that is 0.10 M K^+ . Repeat for concentrations of 100.0 ppm K^+ and 1.0% w/v K^+ .
- KCl
 - K_2SO_4
 - $\text{K}_3\text{Fe}(\text{CN})_6$
- 2-12)** A series of dilute NaCl solutions is prepared, starting with an initial stock solution of 0.100 M NaCl . Solution A is prepared by pipeting 10 mL of the stock solution into a 250-mL volumetric flask and diluting to volume. Solution B is prepared by pipeting 25 mL of solution A into a 100-mL volumetric flask and diluting to volume. Solution C is prepared by pipeting 20 mL of solution B into a 500-mL volumetric flask and diluting to volume. What is the molar concentration of NaCl in solutions A, B, and C?
- 2-13)** What is the molar concentration of NO_3^- in a solution prepared by mixing 50.0 mL of 0.050 M KNO_3 with 40.0 mL of 0.075 M NaNO_3 ? What is p NO_3 for the mixture?

- 2-14)** What is the molar concentration of Cl^- in a solution prepared by mixing 25.0 mL of 0.025 M NaCl with 35.0 mL of 0.050 M BaCl_2 ?
- 2-15)** To determine the concentration of ethanol in cognac a 5.00-mL sample of cognac is diluted to 0.500 L. Analysis of the diluted cognac gives an ethanol concentration of 0.0844 M. What is the molar concentration of ethanol in the undiluted cognac?

Chapter 3: Laboratory Practical Guide



3.1 Introduction

Preparing stock standard solution in laboratory is a critical step which affects the whole result by any lab if miss prepared. Standard preparation should be done by skilled analyst. To be a skilled analyst first you have to make sure understood all calculations related to standard preparation. In chapters one and two we discussed in deeply the best way to make calculations with lots of examples. Those are to help you solving any kind of calculation problems in your laboratory life.

In this chapter we will explain in details about most laboratory skills and techniques to help you preparing your standard in accurate way, we will discuss important skills and common laboratory errors. Reading this section is not enough to start working in lab, you should be supervised by skilled chemist or teacher in the first time before you be able to work independent.

3.2 Basic Laboratory Rules

Safety first, so we have always to get a look at laboratory main rules, following are list of most common rules:

3.2.1 General Rules

1. **Do NOT** work with hazardous substances without a second person being present
2. **Do NOT** eat, drink or smoke in the laboratory under any circumstances
3. **ALWAYS** Keep your working area clean and tidy and free of clutter
4. **ALWAYS** Keep benches tidy and gangways clear
5. **ALWAYS** support gas cylinders, and ALWAYS close cylinder valves after use
6. **ALWAYS** label containers in plain English with the common known name of the substance and the appropriate hazard warning sign
7. **ALWAYS** secure the tops of reagent bottles immediately after use
8. **ALWAYS** work with fume cupboard when dealing with organic solvent or acid or volatile substance
9. **ALWAYS** clear up spillages immediately
10. **Do NOT** leave equipment using water, gas or electricity on overnight

3.2.2 Personal Protection Rules

1. **ALWAYS** wear a lab coat and appropriate eye protection, e.g. safety spectacles, goggles or face shield

2. **ALWAYS** use the appropriate gloves whenever handling chemicals or hazardous substances, and **ALWAYS** check their integrity before use, ensuring they will give you protection against the substance being used
3. **ALWAYS** wear proper footwear, do NOT wear open toed footwear

3.2.3 Hygiene Rules

1. **Do NOT** pipette by mouth
2. **ALWAYS** wash hands after using any substances hazardous to health, on leaving the laboratory and before visiting the toilet
3. **Do NOT** touch surfaces with your contaminated gloves if they may be touched by others (phones, door handles etc)

3.2.4 Emergencies Rules

1. **ALWAYS** know where your nearest fire extinguisher and first aid kit are
2. **ALWAYS** know your emergency escape route and assembly point

3.2.5 Storage and Disposal Rules

1. **ALWAYS** keep broken glassware and sharps separate from other waste and **ALWAYS** dispose of in the appropriate containers
2. **ALWAYS** return stock bottles/jars/dewar's etc of highly flammable liquids or acids to their correct store cupboard after work has finished
3. **Do NOT** have more than 500 ml of a flammable solvent in use at any one time on the bench

3.3 Basic Equipment and Instrumentation

There Measurements in lab are made using appropriate equipment or instruments. The array of equipment and instrumentation used in analytical chemistry is impressive, ranging from the simple and inexpensive, to the complex and costly. The instrumentation used to measure mass and much of the equipment used to measure volume are important to all analytical techniques and are therefore discussed in this section.

3.3.1 Instrumentation for Measuring Mass

An object's mass is measured using a balance. The most common type of balance is an electronic balance in which the balance pan is placed over an electromagnet (Figure 3-1). The sample to be weighed is placed on the sample pan, displacing the pan downward by a force equal to the product of the sample's mass and the acceleration due to gravity. The balance detects this downward movement and generates a counterbalancing force using an electromagnet. The current needed to produce this force is proportional to the object's mass. A typical electronic balance has a capacity of 100–200 g and can measure mass to the nearest ± 0.01 to ± 1 mg. Another type of balance is the single-pan, unequal arm balance (Figure 3-2). In this mechanical balance the balance pan and a set of removable standard weights on one side of a beam are balanced against a fixed counterweight on the

beam's other side. The beam itself is balanced on a fulcrum consisting of a sharp knife edge. Adding a sample to the balance pan tilts the beam away from its balance point. Selected standard weights are then removed until the beam is brought back into balance. The combined mass of the removed weights equals the sample's mass. The capacities and measurement limits of these balances are comparable to an electronic balance.

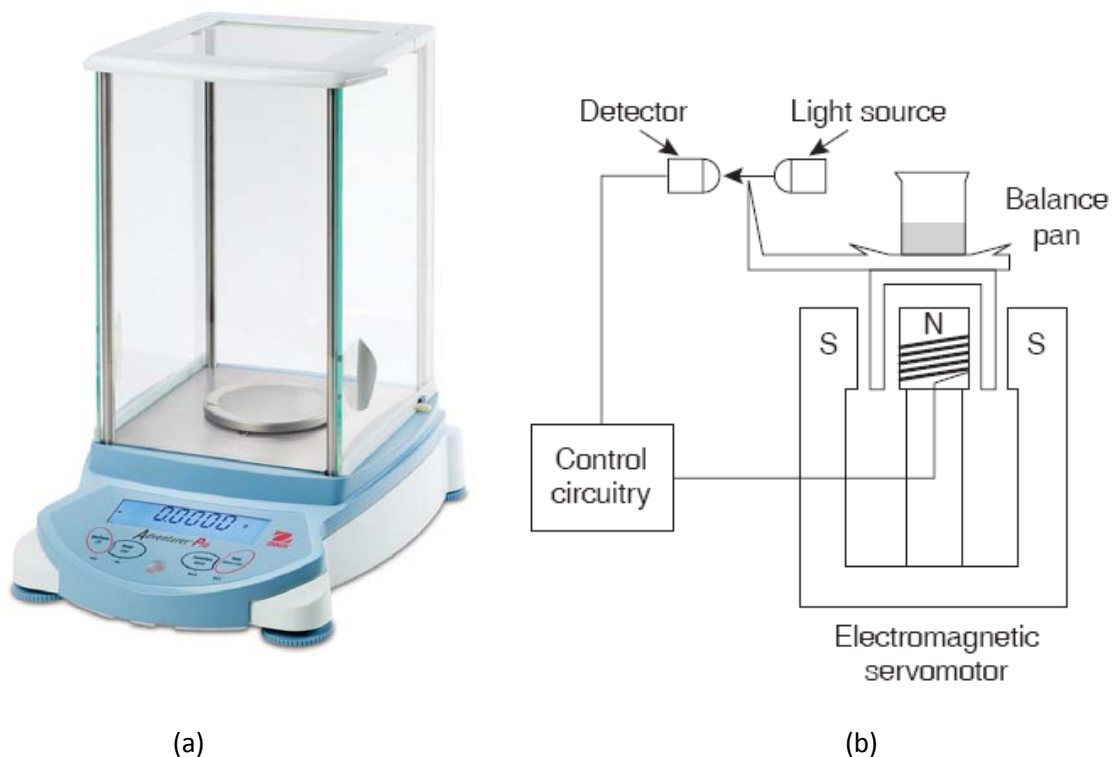


Figure 3-1: (a) Photo of a typical electronic balance. (b) Schematic diagram of electronic balance; adding a sample moves the balance pan down, allowing more light to reach the detector. The control circuitry directs the electromagnetic servomotor to generate an opposing force, raising the sample up until the original intensity of light at the detector is restored.

The mass of a sample is determined by difference. If the material being weighed is not moisture-sensitive, a clean and dry container is placed on the balance. The mass of this container is called the tare. Most balances allow the tare to be automatically adjusted to read a mass of zero. The sample is then transferred to the container, the new mass is measured and the sample's mass determined by subtracting the tare. Samples that absorb moisture from the air are weighed differently. The sample is placed in a covered weighing bottle and their combined mass is determined. A portion of the sample is removed, and the weighing bottle and remaining sample are reweighed. The difference between the two masses gives the mass of the transferred sample.

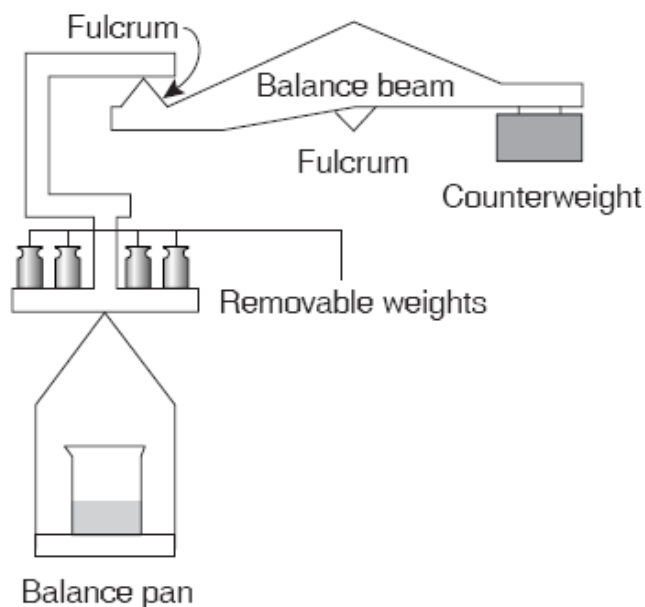


Figure 3-2: Schematic diagram of single-arm mechanical balance.

Several important precautions help to minimize errors in measuring an object's mass. Balances should be placed on heavy surfaces to minimize the effect of vibrations in the surrounding environment and should be maintained in a level position. Analytical balances are sensitive enough that they can measure the mass of a fingerprint. For this reason, materials placed on a balance should normally be handled using tongs or laboratory tissues. Volatile liquid samples should be weighed in a covered container to avoid the loss of sample by evaporation. Air currents can significantly affect a sample's mass. To avoid air currents, the balance's glass doors should be closed, or the balance's wind shield should be in place. A sample that is cooler or warmer than the surrounding air will create convective air currents that adversely affect the measurement of its mass. Finally, samples dried in an oven should be stored in a desiccators to prevent them from reabsorbing moisture from the atmosphere.

3.3.2 Equipment for Measuring Volume

Analytical chemists use a variety of glassware to measure volume, several examples of which are shown in Figure 3-3, 3-4. The type of glassware used depends on how exact the volume needs to be. Beakers, dropping pipettes, and graduated cylinders are used to measure volumes approximately, typically with errors of several percent. Pipettes and volumetric flasks provide a more accurate means for measuring volume. When filled to its calibration mark, a volumetric flask is designed to contain a specified volume of solution at a stated temperature, usually 20 °C. The actual volume contained by the volumetric flask is usually within 0.03–0.2% of the stated value. Volumetric flasks containing less than 100 mL generally measure volumes to the hundredth of a milliliter, whereas larger volumetric flasks measure volumes to the tenth of a milliliter. For example,



Figure 3-3: Beaker

a 10-mL volumetric flask contains 10.00 mL, but a 250-mL volumetric flask holds 250.0 mL (this is important when keeping track of significant figures).

Because a volumetric flask contains a solution, it is useful in preparing solutions with exact concentrations. The reagent is transferred to the volumetric flask, and enough solvent is added to dissolve the reagent. After the reagent is dissolved, additional solvent is added in several portions, mixing the solution after each addition. The final adjustment of volume to the flask's calibration mark is made using a dropping pipette. To complete the mixing process, the volumetric flask should be inverted at least ten times.

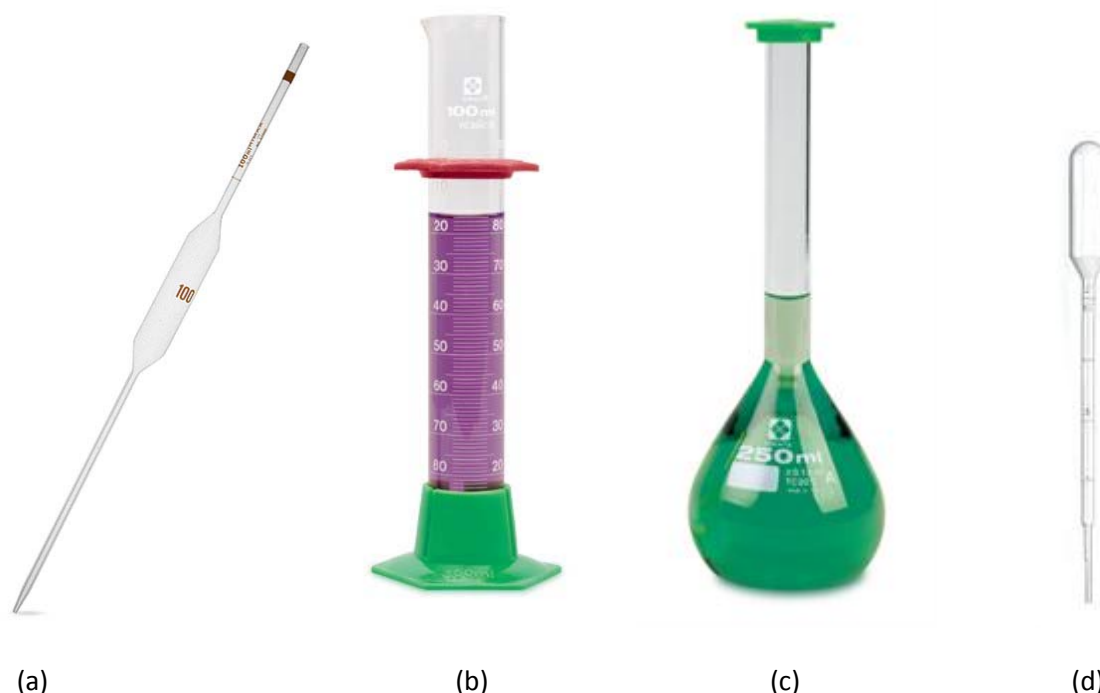


Figure 3-4: Range of widely used volumetric transfer glassware (a) Transfer pipette (b) Graduated cylinder (c) Volumetric flask (d) Dropping pipette.

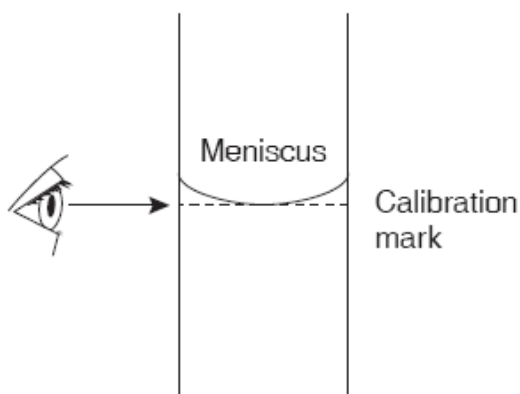


Figure 3-5: Calibration mark for volumetric glassware

A pipette is used to deliver a specified volume of solution. Several different styles of pipettes are available (Figure 3-4). Transfer pipettes provide the most accurate means for delivering a known volume of solution; their volume error is similar to that from an equivalent volumetric flask. A 250-mL transfer pipette, for instance, will deliver 250.0 mL. To fill a transfer pipette, suction from a rubber bulb is used to pull the liquid up past the calibration mark (never use your mouth to suck a solution into a pipette). After replacing the bulb with your finger, the liquid's level is adjusted to the calibration mark, and the outside of the pipette is wiped dry. The pipette's contents are allowed to drain into the receiving container with the tip of the pipette

touching the container walls. A small portion of the liquid remains in the pipette's tip and should not be blown out. Measuring pipettes are used to deliver variable volumes, but with less accuracy than transfer pipettes. With some measuring pipettes, delivery of the calibrated volume requires that any solution remaining in the tip be blown out. Digital pipettes and syringes can be used to deliver volumes as small as a microliter.

Three important precautions are needed when working with pipettes and volumetric flasks. First, the volume delivered by a pipette or contained by a volumetric flask assumes that the glassware is clean. Dirt and grease on the inner glass surface prevents liquids from draining evenly, leaving droplets of the liquid on the container's walls. For a pipette this means that the delivered volume is less than the calibrated volume, whereas drops of liquid above the calibration mark mean that a volumetric flask contains more than its calibrated volume. Commercially available cleaning solutions can be used to clean pipettes and volumetric flasks.

Second, when filling a pipette or volumetric flask, set the liquid's level exactly at the calibration mark. The liquid's top surface is curved into a meniscus, the bottom of which should be exactly even with the glassware's calibration mark (Figure 3-5). The meniscus should be adjusted with the calibration mark at eye level to avoid parallax errors. If your eye level is above the calibration mark the pipette or volumetric flask will be overfilled. The pipette or volumetric flask will be under filled if your eye level is below the calibration mark.

Finally, before using a pipette or volumetric flask you should rinse it with several small portions of the solution whose volume is being measured. This ensures that any residual liquid remaining in the pipette or volumetric flask is removed.

3.4 *Preparing Solutions*

Solutions of known concentration can be prepared in a number of different ways depending on the nature of the analyte and/or the concentration required:

- Weighing out a solid material of known purity, dissolving it in a suitable solvent and diluting to the required volume
- Weighing out a liquid of known purity, dissolving it in a suitable solvent and diluting to the required volume
- Diluting a solution previously prepared in the laboratory
- Diluting a solution from a chemical supplier.

Remember to record all masses and volumes used in the preparation of solutions in a laboratory workbook, and to show how you calculated the concentration of the solution. The procedure for preparing a solution by dissolving a solid material is shown in Figure 3-6. The procedure for preparing a solution by dilution of a more concentrated solution (either prepared in the laboratory or from a chemical supplier) is shown in Figure 3-7.

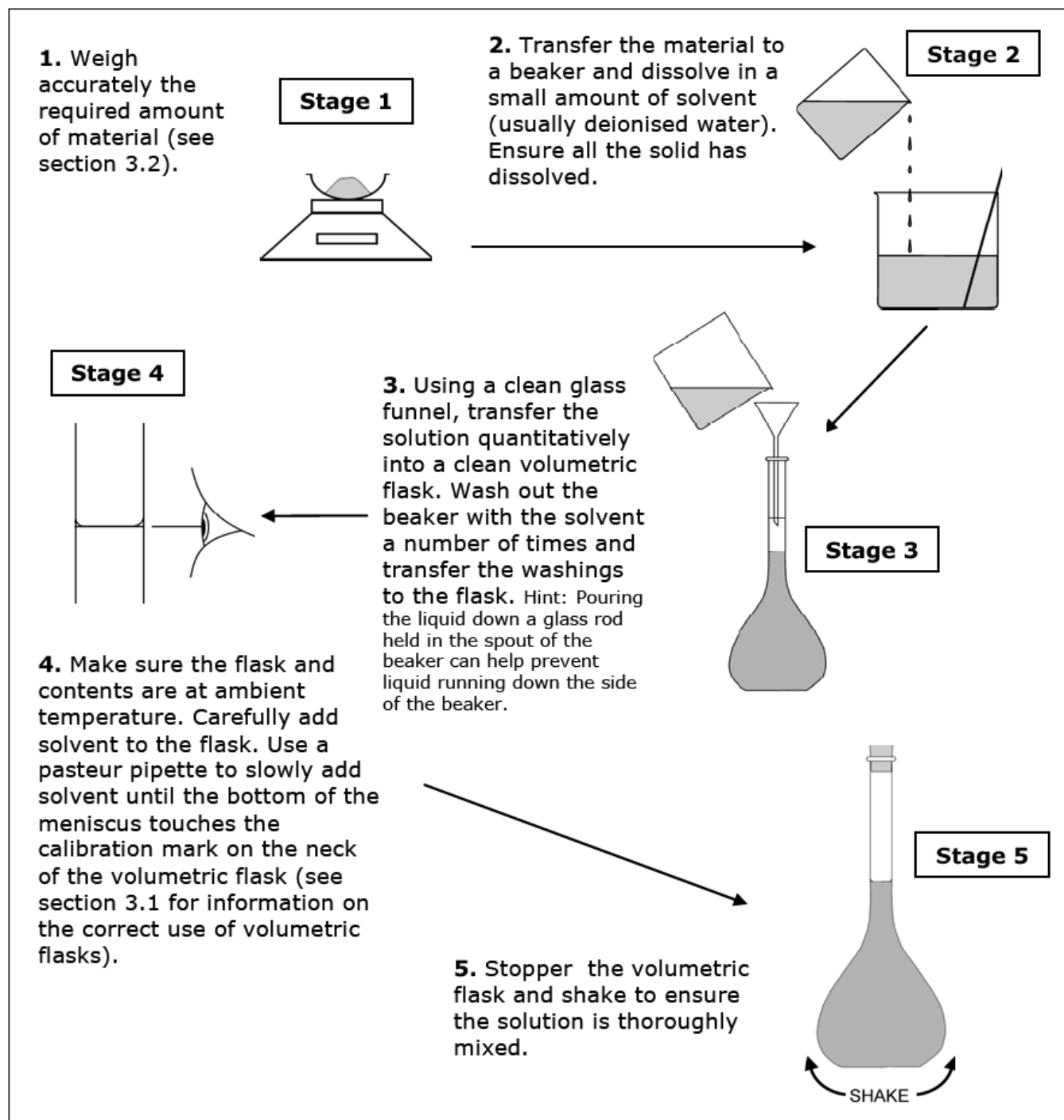


Figure 3-6: Procedure for preparing a solution of known concentration from a known amount of a solid material

Note that in some cases the solute may be a liquid rather than solid. The procedure is very similar to that shown in Figure 3-6. The required amount of the liquid is weighed accurately. The liquid is then transferred directly to the volumetric flask containing some of the solvent.

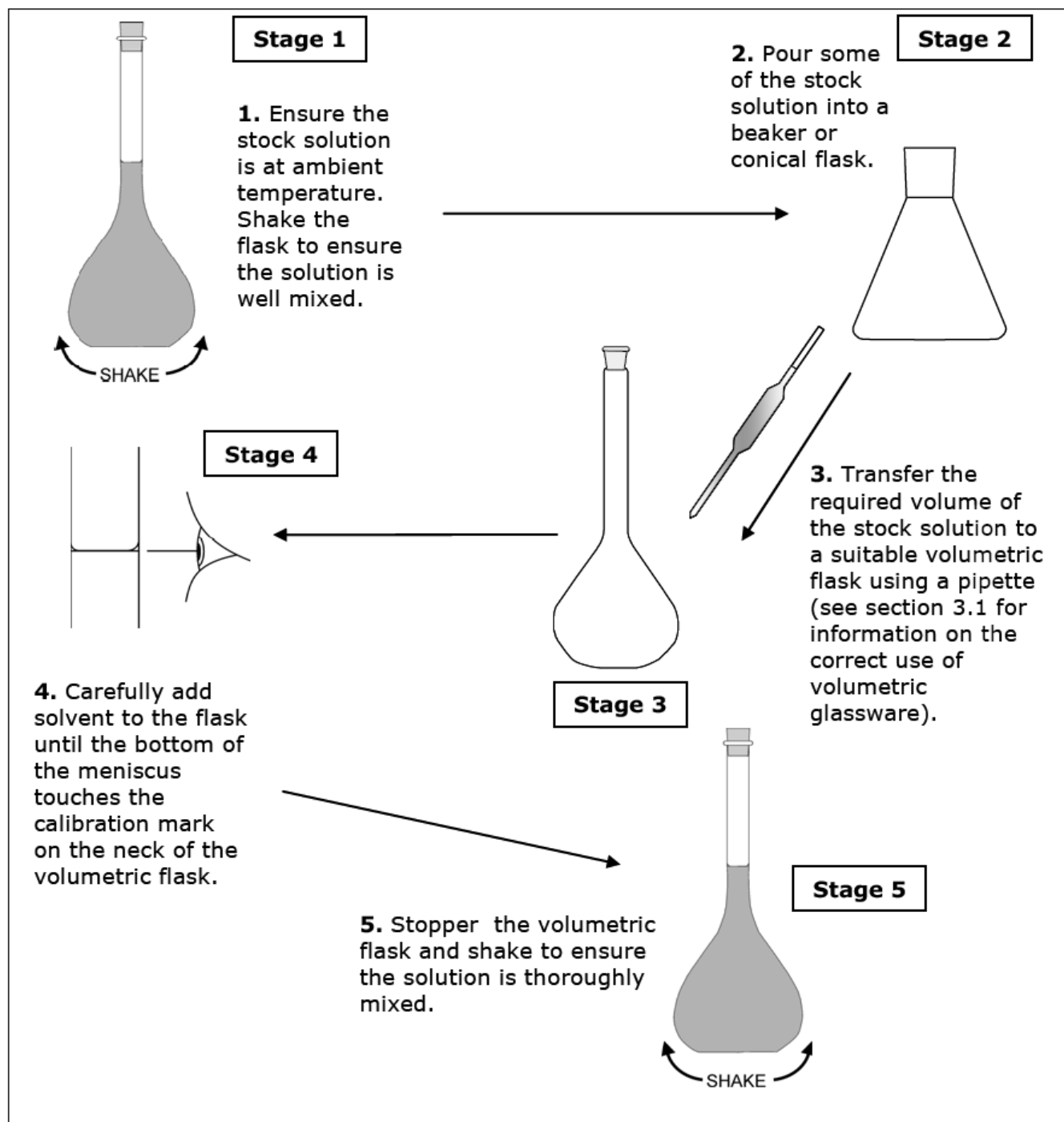


Figure 3-7: Procedure for preparing a solution of known concentration by dilution

3.4.1 Labeling and storing solutions

Once you have prepared the solution you need to think about how you will store it and how it will be identified in the future. Remember the following key points:

- Solutions should not be stored in volumetric flasks – transfer them to a suitable container for storage;
- Ensure that solutions are stored correctly. Some solutions will need to be stored in a refrigerator while others may be light-sensitive and need to be stored in amber bottles;
- All solutions should be clearly labeled with the following information:

- 1- The name and concentration of the solution;
 - 2- Date of preparation;
 - 3- Name of analyst;
 - 4- Review or expiry date;
 - 5- Hazard information (if appropriate);
- The label must be securely attached to the container and be written in water insoluble ink.

In some cases, particularly where volatile solvents are used, it is useful to check for any changes in the mass of the solution during storage. After the solution has been prepared, it is transferred to a suitable container and the mass of the sealed container and the solution is recorded. Prior to an aliquot of the solution being used, the container is re-weighed. The mass should not be significantly different from that recorded prior to the solution being stored. After the required volume of the solution has been transferred from the storage container, the solution is reweighed before being returned to storage. If a significant change in mass is observed after the solution has been stored then it should not be used.

3.4.2 Checklist for preparing solutions of known concentration

Table 3-1 summarizes the key points to remember when preparing solutions of known concentration.

Table 3-1: Do and don't for preparing solutions of known concentration

Do	Don't
Preparing a solution by dissolving a solid material	
■ Use a material with a suitable purity (grade) and ensure that it has been stored correctly and is within its expiry date.	■ Use material that appears to have been stored incorrectly or that has passed its expiry date.
■ Use a clean, dry spatula to transfer the required amount of material.	■ Return unused material to the original bottle.
■ Make sure you have correctly calculated the amount of material required.	
■ Weigh accurately the required amount of material.	
■ Ensure that all glassware used is clean, dust free and undamaged.	■ Use dirty glassware or glassware that is damaged and/or has faded graduation marks.
■ Transfer the material to a beaker and dissolve in a small amount of solvent.	■ Transfer the material directly to the volumetric flask.
■ Make sure that material has dissolved completely.	
■ Use a small glass funnel to transfer the solution from the beaker to the volumetric flask.	
■ Rinse the beaker with solvent and transfer the rinsing to the volumetric flask.	
■ Make sure that the solution is at ambient temperature before making the solution up to	■ Make the solution up to the calibration mark if its temperature is significantly different

Do	Don't
the calibration mark with solvent.	from the ambient temperature. (Note that if the ambient temperature is significantly different from the calibration temperature of the glassware used this will increase the uncertainty in the concentration of the solution.)
■ Make sure the solution is mixed thoroughly before use.	
Preparing solutions by dilution of a stock solution	
■ Make sure the stock solution is at room temperature.	■ Use a stock solution straight from the refrigerator.
■ Plan a dilution scheme to minimize the uncertainty in the concentration of the diluted solution.	■ Carry out large dilutions in a single step.
■ Ensure that all glassware used is clean, dust free and undamaged.	■ Use dirty glassware or glassware that is damaged and/or has faded graduation marks.
■ Ensure the stock solution is well mixed before use.	
■ Transfer the stock solution to a beaker or conical flask for pipetting.	■ Pipette directly from the stock solution bottle/flask.
■ Make sure the diluted solution is well mixed before use.	■ Return unused solution to the stock bottle.
All Solutions	
■ Transfer the solution to a suitable container for storage.	■ Store solutions in volumetric flasks.
■ Clearly label containers with: the name and concentration of the solution; date of preparation; name of analyst; review or expiry data; hazard labels (if appropriate).	
■ Store solutions correctly (e.g. in a refrigerator if necessary).	

3.5 *Laboratory Note Book*

Finally, we cannot end a chapter on the basic tools of analytical chemistry without mentioning the laboratory notebook. Your laboratory notebook is your most important tool when working in the lab, providing a complete record of all your work. If kept properly, you should be able to look back at your laboratory notebook several years from now and reconstruct the experiments on which you worked.

Your instructor will probably provide you with detailed instructions on how he or she wants you to maintain your notebook. Of course, you should expect to bring your notebook to the lab. Everything you do, measure, or observe while working in the lab should be recorded in your notebook as it takes place. Preparing data tables to organize your data will help ensure that you record the data you need and that you can find the data when it is time to calculate and analyze your results. Writing a narrative to accompany your data will help you remember what you did, why you did it, and why you thought it was significant. Reserve space for your calculations, for

analyzing your data, and for interpreting your results. Take your notebook with you when you do research in the library.

Maintaining a laboratory notebook may seem like a great deal of effort, but if you do it well you have a permanent record of your work. Scientists working in academic, industrial, and governmental research labs rely on their notebooks to provide a written record of their work. Questions about research carried out at some time in the past can be answered by finding the appropriate pages in the laboratory notebook. A laboratory notebook is also a legal document that helps establish patent rights and proof of discovery.

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