* Write about determine and indetermine errors. How care determinate errors minimized?

Ans:

The errors which affect our experimental result may be divided into systematic or random determine and random or indetermine errors.

Determine (systematic) errors: These are errors which can be avoided or whose magnitude can be determined. The most important of them are i. operational and personal errors ii. Instrumental and reagent errors iii. Errors of method iv. Additive and proportional errors.

Indetermine (Random) erros: These errors manifest themselves by the slight variations that occur in successive measurment made by the same observer with the greatest care under as nearly identical conditions as possible. They are due to causes over which the analyst has no control and which, in general are so intangible that they are incapable of analysis.

Systematic or determine errors can be reduced by a no. of method:

(i) Calibration of apparatus and application of Corrections: All instruments (weight, flasks, burettes pipettes etc) should be calibrated and the appropriate Corrections applied to the Original measurments. In some cases where an error cannot be eliminates it is possible to apply a Correction for the effect that it produces.

(ii) Running a blank determination: This consists of in carrying out a separate determination the sample being omitted, Under exactly the same experimental conditions as are employed for the actual analysis of the sample. The object is to find out the effect of the impurities introduced through the reagents of vessels, or to determine the excess of standard solution necessary to establish the end-point Under the conditions met with for the

titration of the unknown sample. (iii) Running control determination using primary standard. (iv) use independent method of analysis (v) Running parallel determinations. (vi) Standard addition (vii) Internal Standards (viii) Implication methods

(*) High degree of precision may not imply accuracy." Justify.

Ans

Accuracy refers to the closeness of a measured value to a standard or known value. For example, if in lab, one obtains a weight measurement of 3.2 kg for a given substance, but the actual or known weight is 10 kg, then the measurement is not accurate. In this ~~measurement~~ case, measurement is not close to known value.

On the other hand precision refers to the closeness of two or more measurements to each other. Using the example above, if one weigh a given substance five times, and get 3.2 kg each time, then the measurement is very precise. Precision is independent of accuracy. So from above example it is clear that high degree of precision may not imply accuracy.

*

What do you mean by relative error and absolute error?

When a quantity is measured even with extreme care, it is found that its results of successive determinations differ among themselves. The average value is accepted as the most probable. This may not always be the true value.

The absolute error of a determination is the difference between the observed (or measured) value and the true value of the quantity measured. It is a measure of the accuracy of the measurement.

The relative error is the absolute error divided by the true value. It is usually expressed in term of percentage.

The true or absolute value of a quantity can not be established experimentally, so that the observed result must be compared with the most probable value.

If we consider a series of observation (n) having magnitude of the quantity $x_1, x_2, x_3 \cdots x_n$, then

$$\text{Average (or most probable value)} = \frac{x_1 + x_2 + \cdots x_n}{n} = \frac{\sum x_i}{n}$$

For the measurment

$$\text{Absolute error} = (\bar{x} - x_1)$$

$$\text{Relative error} = \left(\frac{\bar{x} - x_1}{x_1} \times 100\right)\%$$

* What are accuracy and Precision?

Accuracy refers to the closeness of a measured value to a standard or known value. For example, if in lab you obtained a weight measurment of 3.2 kg for a given substance, but the actual or known weight is 10 kg, then the measurment is not accurate. In this case the measurment is not close to the known value.

Precision refers to the closeness of two or more measurements to each other. Using the example above, if anyone weight a given substance five times, and get 3.2 kg each time, then the measurment is very precise but inaccurate. Precision is independent of accuracy. One can be very precise but inaccurate, as described and one can also be accurate but imprecise.

For example, if on average, one's measurment for a given substance are close to the known value, but the measurements are far from each other, then he/she have accuracy without precision.

*

In a set of measurements, the following concentration of iron (in ppm) were reported.

9.98, 9.99, 9.98, 9.95, 10.00, 10.20

Calculate (i) Standard deviation
(ii) Coefficient of variation

Ans:

x	$\bar{x}$	$(x_i - \bar{x})$	$(x_i - \bar{x})^2$
9.98	10.01	−0.03	0.0009
9.99	10.01	−0.02	0.0004
9.98	10.01	−0.03	0.0009
9.95	10.01	−0.06	0.0036
10.00	10.01	−0.01	0.0001
10.20	10.01	0.19	0.0361
			0.042

$\therefore$ variance $(s) = \frac{\sum (x_i - \bar{x})^2}{n-1} = \frac{0.042}{05} = 0.084$

(i) Standard deviation $(\sigma) = \sqrt{0.084}$
$= 0.2898$

(ii) Coefficient of variation $= \frac{\sigma}{\bar{x}} \times 100\%$
$= \frac{0.2898}{10.01} \times 100\%$
$= 2.895\%$.

* Zinc Contents of a brass sample are analyzed with the following results:

Zn: 33.37%, 33.27%, 33.34% and 33.30%.

Calculate Standard deviation and coefficient of variation of the ~~sample~~ analysis.

Ans.

x	$\bar{x}$	$(x_i - \bar{x})$	$(x_i - \bar{x})^2$
33.37	33.32	0.05	0.0025
33.27	33.32	-0.05	0.0025
33.34	33.32	0.02	0.0004
33.30	33.32	-0.02	0.0004
			0.00145

$\therefore$ Variance $= \frac{\sum (x_i - \bar{x})^2}{n-1} = \frac{0.00145}{4-3} = 0.000483$

i. Standard deviation $= \sqrt{0.000483}$

$= 0.0219$

ii. Coefficient of variation $= \frac{0.02198}{33.32} \times 100$

$= 0.065\%$

* Describe briefly about the possible source of errors in quantitative analysis?

Ans The possible sources of errors are:

i. Operational and personal errors: These are due to factors for which the individual analyst is responsible and are not in any way connected with the method or procedure: they form part of the personal equation of the observer. The errors are mostly physical in nature and occur when sound analytical is not followed.

Personal errors may arise also because, some persons are unable to judge colour change sharply in visual titration, which may result in a slight overstepping of the end point.

ii. Instrumental and reagent errors: This arise from the faulty construction of balance, the use of uncalibrated or improperly calibrated weight, graduated glassware, and other instruments; the attack of reagent upon glassware, porcelain etc., resulting in the introduction of foreign materials, volatilisation of platinum at very high temperature and the use of reagents containing impurities.

iii Errors of method: These originate from incorrect sampling and from incompleteness of a reaction. In gravimetric analysis errors may arise owing to appreciable solubility of ppt precipitates, co-precipitation and post-precipitation of substance other than intended ones.

iv Additive and proportional errors: The absolute value of additive error is independent of the amount of the constituent present in the determination. Examples of additive errors are loss in weight of a crucible in which a ppt. is ignited, and errors in weights.

v Random errors: These errors manifest themselves by the slight variations that occur in successive measurements made by the same observer with the greatest care under as nearly identical condition as possible. They are due to cause over which the analyst has no control and which are in general so intangible that they are incapable of analysis.

* The molar concentrations of a particular solution were found to be 0.2041, 0.2049, 0.2039 and 0.2043 in four separate experiments. Find out the value of mean, median, range, average deviation, relative average deviation, Standard deviation and the coefficient of variance.

Ans

For the even values of experiments:

0.2039, 0.2041, 0.2043, 0.2049

$$\text{Median (M)} = \frac{0.2041 + 0.2043}{2}$$

$$= 0.2042$$

$$\text{Arith. Mean} = \frac{0.2041 + 0.2049 + 0.2039 + 0.2043}{4}$$

$$= 0.2043$$

$$\text{Range, R} = 0.2049 - 0.2039 = 0.001$$

$$\text{Average deviation } \bar{d} = \frac{0.0002 + 0.0006 + 0.0004 + 0.0}{4}$$

$$= 0.0003$$

$$\text{Relative average deviation} = \frac{\bar{d}}{\bar{x}} \times 1000$$

$$= \frac{0.0003}{0.2043} \times 1000 = 1.5$$

$$\text{Standard deviation. } S = \sqrt{\frac{(0.0002)^2 + (0.0006)^2 + (0.0004)^2 + 0^2}{4-1}}$$

$$= 0.0004$$

$$\text{Coefficient of variance } C_v = \frac{S \times 100}{\bar{x}} = \frac{0.0004 \times 100}{0.2043} \text{ f.}$$

$$= 0.2 \text{ f.}$$

* The Percentage of a Constituent A in ram ore were found to be 48.32, 48.35, 48.24, 48.18 and 48.39. Calculate mean deviation and standard deviation.

Ans

x	$\bar{x}$	$x_i - \bar{x}$	$(x_i - \bar{x})^2$
48.32	48.296	0.024	0.000576
48.35	48.296	0.054	0.002916
48.24	48.296	−0.056	0.003136
48.18	48.296	−0.116	0.01345
48.39	48.296	0.094	0.00883
			0.01142

$$\text{Variance} = \frac{\sum (x_i - \bar{x})^2}{n-1} = \frac{0.01142}{4}$$

$$= 0.002856$$

$$\text{Standard deviation} = \sqrt{0.002856}$$

$$= \underline{0.0534}$$

$$\text{mean deviation} = \frac{0.01142}{5}$$

$$= \underline{0.0022}$$

* What are mean and median?

The arithmatic mean or average is obtained by adding together all the results of different determinations, & dividing the sum by the no. of determination. It is denoted as $\bar{x}$

$$\bar{x} = \frac{x_1 + x_2 \cdots + x_n}{n}$$

$$= \frac{\sum_i x_i}{n}$$

The median is the average or middle values of in a set. for an odd number of values, the middle value is the median, & for an even no. of values, the average of the two middle values is the median. This median is more likely to be closer to the tone value than the mean, because a single bad result may interfere the mean more than the median.

For the odd value 18, 19, 20, 21, & 22 the median = 20

For the even values 18, 19, 20, 21, 22 & 23, the median is $\frac{20+21}{2} = 20.5$

*

Mode: The mode is the frequency or no. of occasions upon which a given value appears in the data. For example for a set of observations 5, 4, 6, 6, 4, 5, 6, 6, 3, the mode is 6 and the frequency of the value is, 4.

Deviation: The difference between any result of an analysis and the mean (or average) of a series of results is called the deviation from the mean. The deviation is positive if the value is greater than the mean, and negative if it is lows than the mean. It is denoted by d.

Average deviation: It is the average of the absolute value of deviations in a series of determinations. Its relative magnitude is indicative of the precision of a series of determinations. The smaller the average deviation, the greater is the precision. The average deviation is represented as $\bar{d}$ and given the formula.

$$\bar{d} = \frac{d_1 + d_2 + d_3 + \cdots d_n}{n} = \frac{1}{n}\sum_i d_i$$

Standard deviation:

When there are four or more determinations we often use the term standard deviation instead of average deviation. The standard deviation carries more significance than the average deviation. The standard deviation is represented as S is given by

$$S = \sqrt{\frac{d_1^2 + d_2^2 + \cdots d_n^2}{n-1}}$$

Range: The range is the arithmetical difference between smallest & the largest values of a series.

Variance: The coefficient of variance is an accurate measure of the precision & is given by

$$C_V = \frac{S \times 100}{\bar{x}}$$

* The tin and zinc contents of a brass sample are analyzed and the following results are obtained:

i. tin percentage: 0.022%, 0.025% and 0.024%.

ii. zinc percentage: 33.27%, 33.37% and 33.34%.

Calculate standard deviation and coefficient of variation for each analysis?

* do yourself

* Define confidence limit, confidence level and confidence interval with suitable examples.

A confidence interval (CI) is a type of interval estimate, computed from the statistics of the observed data, that might contain the true value of unknown population parameter.

Confidence level is ~~the~~ quantifies the level of confidence that the deterministic parameter is captured by the interval. More strictly speaking, the confidence level represents the frequency of possible confidence intervals that contain the true value of the unknown population parameter.

To calculate the confidence limits for a measurement variable, multiply the standard error of the mean times the appropriate t-value. The t-~~ratio~~ value is determined by the probability and the degree of freedom (n-1).

Solvent extraction

* What is solvent extraction? State the advantage of this technique? 2+2

Liquid-Liquid extraction is a separation technique, here a aqueous solution is brought into contact with a second solvent usually organic, immisible with water in order to bring about a transfer of one or more of the solute into the second solvent. The separation are clean, rapid and convenient. The technique is applicable to trace level or large amount of materials. The term solvent extraction can also refer to the separation of a substance from a mixture by preferentially dissolving that substance in a suitable solvent.

Advantage of solvent extraction:

i. A soluble compound is separated from

an insoluble compound or a complex matrix.

ii. From a hydrometallurgical perspective solvent extraction is extensively used in separation and purification of Uranium and plutonium, Zirconium and hafnium, separation of cobalt and nickel etc. Its greatest advantage being its ability to selectively separate out even very similar metal.

iii. It is also widely used in the production of fine organic compounds, the processing of perfumes, the production of vegetable oils and biodiesel.

* What are the differences between distribution ratio and distribution coefficient?

If a solute is added to a system containing two immiscible liquids 1 & 2 (or slightly miscible liquid) and if the solute is soluble in both liquids, then it distributes itself between the two liquids in a definite manner, such that

$$\frac{C_1}{C_2} = K_D$$

where C_1 and C_2 are equilibrium molar concentration of the solute in the two liquids 1 & 2 respectively. K_D is a constant known as the distribution coefficient or partition coefficient. The value of K_D depends only on the temperature of the system and is independent of the relative amount of the two layers and also of the solute.

On the other hand distribution ratio is defined as the total concentration of a solute in the stationary phase in all possible chemical forms over the total concentration of the solute in the mobile phase in all its possible chemical forms.

* What are the ~~difference~~ between distribution ratio and distribution coefficient?

For a solute 'A' to be distributed between two immiscible phases a and b, the Nernst distribution law states that,

$$\frac{\text{Concentration of Solute in Solvent a}}{\text{Concentration of solute in Solvent b}} = \frac{[A]_a}{[A]_b} = K_D$$

K_D is called distribution or partition coefficient.

The law is valid provided the molecular state of solute in the same in both the liquids and temperature is a constant, in other words the distributing species should not undergo dissociation or association in either phase.

The law does not take into account the activities of the various species and hence apply applies only to very dilute solution where the ratio of activities approaches Unity.

In practical applications, the fraction of the total solute in one or other phase is important not if the solute would undergo dissociation or association or interaction with other dissolved species. It is more convenient to introduce the term distribution ratio D (or extraction coefficient E)

$$D = \frac{(C_A)_a}{(C_A)_b}$$

C_A is the concentration of A in all its forms as determined analytically.

* What is known as 'separation factor' in solvent extraction?

If a solution contains two solutes A and B, it often happens that under the conditions favouring the complete extraction of A, some B is extracted as well. The effectiveness of separation increases with the magnitudes of the individual distribution ratio as follows:

$$\beta = \frac{(C_A)_1 / (C_B)_1}{(C_A)_2 / (C_B)_2} = \frac{(C_A)_1}{(C_A)_2} \times \frac{(C_B)_2}{(C_B)_1} = \frac{D_1}{D_2}$$

(β is separation factor and D_1 and D_2 are the distribution coefficients of A and B respectively (in the two solvents 1 & 2)

* Mathematically prove that multi-step solvent extraction has higher efficiency than single-step solvent extraction, using the same volume of extracting solvents. 4 #

Ans

Let W be the amount of solute present in volume V_1 of the solvent 1 (V_1 volume of the solution) and let the volume V_2 of the solvent 2 be added each time till the entire solvent 2 is exhausted. The solvent 2 is added n times, we have

Total volume of the solvent 2 used for extraction $= nV_2$

If W_1 is the amount of the solute that remains unextracted, in solvent 1 after the addition of first lot of V_2 of solvent 2, then

$$\frac{C_1}{C_2} = \frac{W_1/V_1}{(W-W_1)/V_2} = K_D$$

$$\Rightarrow \frac{W_1}{W-W_1} = \frac{K_D V_1}{V_2}$$

$$\Rightarrow \frac{W}{W_1} = \frac{V_2}{K_D V_1} + 1 = \frac{V_2 + K_D V_1}{K_D V_1}$$

$$\Rightarrow \frac{W_1}{W} = \frac{K_D V_1}{(K_D V_1 + V_2)}$$

$$\Rightarrow W_1 = \left(\frac{K_D V_1}{K_D V_1 + V_2}\right) W \quad \text{--- (1)}$$

If W_2 is the amount of the solute remaining unextracted after the addition of 2nd lot, then

$$\frac{C_1'}{C_2'} = \frac{W_2/V_2}{(W_1 - W_2)/V_2} = K_D$$

$$W_2 = \left(\frac{K_D V_1}{K_D V_1 + V_2}\right) W_1$$

$$= \left(\frac{K_D V_1}{K_D V_1 + V_2}\right)^2 W \quad \text{---- (2)}$$

Carrying out the above process n times, we have

$$W_n = \left(\frac{K_D V_1}{K_D V_1 + V_2}\right)^n W \quad \ldots\ldots (3)$$

Thus, the fraction of the solute remaining unextracted in solvent 1 is:

$$\frac{W_n}{W} = \left(\frac{K_D V_1}{K_D V_1 + V_2}\right)^n \quad \ldots (4)$$

If the extraction were carried out in a single stage by adding whole of the solvent 2 in one lots then we would have,

$$\frac{W'}{W} = \left(\frac{K_D V_1}{K_D V_1 + nV_2}\right) \quad \ldots\ldots (5)$$

W' is the amount remaining unextracted

From eqn. (4)

$$\frac{W}{W_n} = \left(\frac{K_D V_1 + V_2}{K_D V_1}\right)^n = \left(1 + \frac{V_2}{K_D V_1}\right)^n$$

$$= 1 + \frac{nV_2}{K_D V_1} + \frac{n(n-1)}{2}\left(\frac{V_2}{K_D V_1}\right)^2 + \ldots \quad (6)$$

From eqn (5)

$$\frac{W}{W'} = \left(\frac{K_D V_1 + nV_2}{K_D V_1}\right) = 1 + \frac{nV_2}{K_D V_1} \quad \ldots\ldots 7$$

Substituting eqn (7) in eqn (6)

$$\frac{W}{W_n} = \frac{W}{W'} + \frac{n(n-1)}{2}\left(\frac{V_2}{K_D V_1}\right)^2 + \ldots\ldots$$

$$\Rightarrow \frac{W}{W_n} > \frac{W}{W'}$$

$$\Rightarrow W_n < W'$$

i.e. the amount of solute remaining unextracted is less in multistep extraction. In other words, the amount of extracted solute is large in multistep extraction than in single stage extraction.

* The distribution coefficient of a substance between chloroform and water is 6.0. Show that amount of caffeine extracted from 100 ml of an aqueous soln is more by using 50 ml chloroform at a time twice than using 100 ml of it at a time.

~~to it yourself~~

* Isobutyric acid has Kd value 3 for diethyl ether and water system. 4.0 gm isobutyric acid is added (M.W. of isobutyric acid is 176) to 350 ml diethyl ether and 100 ml water.

i. Calculate the amount of acid in each layer after equilibrium.

ii. Calculate the percentage of extraction at the first extraction with 100 ml of water.

Ans

$$K_d = \frac{\text{Concentration of isobutyric acid in ether}}{\text{Concentration of isobutyric acid in water}} = 3$$

When the whole 350 ml of ether is used at a time of extraction, suppose w_1 gm of solute pass into ether layer and w_2 gm are left in aqueous layer, so that,

$$\frac{\frac{w_1}{350}}{\frac{w_2}{100}} = 3$$

$$\text{or } \frac{w_1}{w_2} \times \frac{2}{7} = 3$$

$$\text{or } \frac{w_1}{w_2} = \frac{21}{2} = 10.5$$

$w_1 = 10.5 w_2$ or $\frac{w_1}{w_1 + w_2} = \frac{10.5 w_2}{11.5 w_2} = \frac{105}{115}$

91.3% of isobutyric acid originally present.

" Discuss the effect of pH on the solvent extraction of metal chelates.

One of the most important application of solvent extraction is the separation of the metal ion. Metal ion don't tend to dissolve appreciably in the organic layer. To make it soluble into organic solvent its charge must be neutralised. for this organic chelation agent are used to form uncharged complexes that are soluble in Organic solvent.

Step-1: The chelating agent is added to organic layer. It destributes between organic and aqueous layer.

$$[HR]_{org} \rightleftharpoons [HR]_{aq}$$

$$K_{d(HR)} = \frac{[HR]_{org}}{[HR]_{aq}} \quad \text{---- (I)}$$

Step-2: Chelating agent in aqueous solution ionises

$$(HR)_{aq} = H^+_{aq} + R^-_{aq}$$

$$K_a = \frac{[H^+]_{aq}\,[R^-]_{aq}}{[HR]_{aq}} \quad \text{---- (II)}$$

Step-3: The metal ion chelate with the reagent anion to form the uncharged molecule.

$$M^+_{(aq)} + R^-_{(aq)} = MR_{(aq)}$$

$$K_f = \frac{[MR]_{aq}}{[M^+]_{aq}\,[R^-]_{aq}} \quad \text{---- (III)}$$

Fourth Step: The chelate distributes between organic and aqueous phase:

$$[MR]_{aq} \rightleftharpoons [MR]_{org}$$

$$K_d(MR) = \frac{[MR]_{org}}{[MR]_{aq}} \quad \text{--- (10)}$$

$$\text{Distribution ratio } (D) = \frac{[MR]_{org}}{[M^+]_{aq} + [MR]_{aq}}$$

Since $[MR]_{aq}$ is very low so distribution ratio can be written as,

$$D = \frac{[MR]_{org}}{[M^+]_{aq}} \quad \text{--- (V)}$$

From eq (IV) we get,

$$[MR]_{org} = K_d(MR) \cdot [MR]_{aq} \quad \text{--- (VI)}$$

From eq (III), we get

$$[MR]_{aq} = K_f [M^+]_{aq} [R^-]_{aq} \quad \text{--- (VII)}$$

$$[MR]_{org} = K_d(MR) \cdot K_f \cdot [M^+]_{aq} [R^-]_{aq}$$

From equn (II) we get

$$[R^-]_{aq} = \frac{K_a \cdot [HR]_{aq}}{[H^+]_{aq}}$$

$$[MR]_{org} = K_d(MR) \cdot K_f [M^+]_{aq} \cdot \frac{K_a [HR]_{aq}}{[H^+]_{aq}}$$

$$= K_d(MR) \cdot K_f [M^+]_{aq} \cdot \frac{K_a [HR]_{org}}{K_d(HR) \cdot [H^+]_{aq}}$$

$$\therefore D = K_d(MR) \cdot K_f [M^+]_{aq} \cdot \frac{K_a [HR]_{org}}{K_d(HR) \cdot [H^+]_{aq}} \cdot \frac{1}{[M^+]_{aq}}$$

$$= K' \cdot \frac{[HR]_{org}}{[H^+]_{aq}}$$

$$\log D = \log K' + \log [HR]_{org} + pH$$

Thus with increasing f^{11} the log D increases and as a consequence D increase i.e. distribution ration increases.

Chromatography

* Discuss the in brief the basic principles of Chromatography.

Chromatography now refers to any of a diverse group of techniques that effect a separation through a distribution of sample between two immiscible phases. The requirement to distinguished Chromatography from other separation technique is that one phase be Stationary while the second phase be mobile and percolating through the first phase resulting in selective retention of the components of a mixture by the Stationary phase. The Stationary phase which may be solid or liquid or may consists of a mixture of a solid and liquid, is finely divided and is fixed in place. The mobile phase, which may be liquid or gaseous, fills the interstices of the Stationary phase and is able to flow through the Stationary phase. The physical states of the mobile and stationary phases give rise to four basic type of Chromatography

i. liquid-solid Chromatography (LSC)
ii. liquid-liquid " (LLC)
iii. Gas-liquid " (GLC)
iv. Gas-solid " (GSC)

Solid Stationary phase also gives rise to ion exchange Chromatography. Separation of the components, or solutes of a sample results from differences in their rate of adsorption.

*

What are the difference between column chromatography and thin layer chromatography?

In chromatography, separating the complex mixtures of different compounds into individual compounds is significant tools. Sometime a reaction can produce a degradants product other than the desired product, or after analyzing reaction products, a component of the mixture can require being separate after the reaction is completed. Of all the methods to separate, thin layer chromatography and column chromatography is some of the most useful. Thin layer chromatography is commonly utilized for separating the number of mixes in a sample just as their relative polarities rather than physically separating them, which can be practiced with column chromatography.

The main difference are:

i. TLC has a stationary phase of alumina or silica gel. Column chromatography is packed with its stationary phase with an appropriate sorbent material, such as silica.

ii. TLC is carried out against gravity. Column chromatography is run under gravity.

iii. TLC uses for the analytical purpose. Column chromatography uses for the preparative purpose

iv. Column chromatography take more time to separate than the TLC

v. TLC needs less quantity of solvent to separate the analytes. Column chromatography required more amount of solvent.

vi. TLC needs more polar solvent compared to the column chromatography.

* Give a qualitative idea of gas-liquid chromatography?

GLC is a common type of chromatography used in analytical chemistry for separating and analyzing compounds that can be vaporized without decomposition. Typical uses of GLC include testing the purity of a particular substance, or separating the different components of a mixture. In preparative chromatography GLC can be used to prepare pure compounds from a mixture.

In GLC, the mobile phase is carrier gas, usually an inert gas such as helium or an unreactive gas such as nitrogen. Helium remains the most commonly used carrier gas in about 90% of instruments although hydrogen is preferred for improved separations. The stationary phase is a microscopic layer of liquid on an inert solid support, inside a piece of glass or metal tubing called a column.

The gaseous compounds being analyzed interact with the walls of the column, which is coated with a stationary phase. This causes each compound to elute at a different time, known as the retention time of the compound.

Gas chromatography is in principle similar to column chromatography, but has several notable differences. First, the process of separating the components in a mixture is carried out between a liquid stationary phase and a gas mobile phase, whereas in column chromatography the stationary phase is solid and mobile phase is a liquid. Second, the column through which the gas phase passes is located in an oven where the temperature of the gas can be controlled, whereas column chromatography has no such temperature control. Finally, the concentration of a compound in the gas phase is solely function of the vapor pressure of the gas.

Discuss the principle and technique of thin-layer chromatography?

Technique

Thin-layer chromatography technique used to separate non volatile mixtures. Thin-layer chromatography is performed on a sheet of glass, plastic or aluminium foil, which is coated with a thin layer of adsorbent material, usually silica gel, aluminium oxide. This layer of adsorbent is known as the stationary phase.

After the sample has been applied on the plate, a solvent or solvent mixture is drawn up the plate via capillary action. Because different analytes ascend the TLC plate at different rates, separation is achieved. For example, with silica gel, a very polar substance, non-polar mobile phases such as heptane are used.

After the experiment, the spots are visualized. often this can be done simply by projecting ultraviolet light onto the sheets the sheets are treated with a phosphor, and dark spots appear on the sheet where compounds absorb light impinging on a certain area.

To quantify the results, the distance traveled by the substance being considered is divided by the total distance traveled by the mobile phase. This ratio is called retardation factor (R_f). In general, a substance whose structure resembles the stationary phase will have low R_f, while one that has a similar structure to the mobile phase will have high retardation factor.

Principle

Different compounds in the sample mixture travel at different rates due to the differences in their attraction to the stationary phase and because of differences in solubility in the solvent. By changing the solvent, or perhaps using a mixture, the separation of components can be adjusted. Chemists often use TLC to develop a protocol for separation by chromatography and use TLC to determine which fractions contain the

desired compounds. Separation of compound is based on the competition of the solute and the mobile phase for binding sites on the stationary phase. For instance, if normal-phase silica gel is used as the stationary phase, it can be considered polar. Given two compounds that differ in polarity, the more polar compound has a stronger interaction with the silica and is, therefore, better able to displace the mobile phase from the available binding sites. As a consequence, the less polar compound moves higher up the plate.

* Define Rf value.

The Rf value is defined as the ratio of the distance moved by the solute and the distance moved by the solvent

Rf is the 'retardation factor' which is the ratio of the distance traveled by a compound in a mobile phase compared with the distance traveled by the front of the mobile phase itself. It is always greater than or equal to zero, and less than or equal to 1.

$$Rf = \frac{\text{migration distance of substance}}{\text{migration distance of solvent front}}$$

* Explain the term 'column adsorption chromatography'.

Column chromatography in chemistry is a chromatography method used to isolate a single chemical compound from a mixture. Chromatography is able to separate substances based on differential adsorption of compounds to the adsorbent; compounds move through the column at different rates, allowing them to be separated into fractions. The technique is widely applicable, as many different adsorbents can be used with a wide range of solvents. ~~The technique is widely applicable, as many different adsorbents can~~ be used on scales from micrograms up to kilograms. The main advantage of column chromatography is the relatively low cost and disposability of the stationary phase used in the process. The latter prevents cross-contamination and stationary phase degradation between recycling. Column chromatography can be done using gravity to move the solvent, or using compressed gas to

push the solvent through the column.

* Explain the terms elution and eluent?

In a liquid chromatography experiment, for example, an analyte is generally adsorbed, or bound to an adsorbent in a liquid chromatography column. The adsorbent, a solid phase, is a powder which is coated onto a solid support. Based on an adsorbent's composition, it can have varying affinities to hold onto other molecules - forming a thin film on its surface. Elution then is the process of removing analytes from the adsorbent by running a solvent, called an eluent, past the adsorbent/analyte complex.

The eluent is the "carrier" portion of the mobile phase. It moves the analytes through the chromatograph. In liquid chromatography, the eluent is the liquid solvent; in gas chromatography, it is the carrier gas.

The eluate is the analyte material that emerges from the chromatograph. It specifically includes the analytes and solutes passing through the column, while the eluent

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How are non volatile inorganic salts used in gas chromatography?