# EXPERIMENTAL TECHNIQUES IN CHEMISTRY

## DEFINITIONS

*(May be used in short questions with examples)*

(1) **Analysis:**

The process of determination of composition of a substance quantitatively or qualitatively is called analysis. The analysis can be volumetric or grammetric. Acid-base titration is quantitative while salt analysis is qualitative analysis.

(2) **Analyte:**

That sample of the substance which is being analysed is called analyte. Acid or base whose molality is being determined by volumetric analysis is analyte.

(3) **Analytical chemistry:**

That branch of chemistry which deals with the quantitative and qualitative analysis is called analytical chemistry. Quantitative analysis is volumetric and grammetric. Qualitative analysis is salt analysis.

(4) **Chromatographic tank:**

That vessel in which the mobile phase is stored is called chromatographic tank.

(5) **Crystal:**

It is a discrete solid particle which is bounded by definite faces. These faces intersect at definite angles and show certain symmetry characteristics. Unit cell represents the picture of a crystal.

(6) **Crystallization:** **(Guj. 2008, F. Abad 2008, Lahore 2014)**

It is the removal of a solid from solution by increasing its concentration above the saturation point in such a way that the excessive solid separates out in the form of crystals. The crystalline substance is though to be pure. Sugar, we eat is in the form of crystals.

(7) **Distribution coefficient:**

It is a constant which is a ratio of concentration of a solute in two solvents under the given conditions. It is a constant quantity under the given conditions for a system.

(8) **Distribution law:** **(D. G. Khan 2012, D.G. Khan 2014, Rwp. 2014, B. Pur 2014)**

According to this law a substance is distributed between two immiscible liquids in such a way that its concentration in two liquids is constant. It is independent of the amount of the solvent added. It gives distribution coefficient and is represented by $K_D$.

(9) **Filter medium:** **(Rwp. 2012)**

That porous material which is used for filtration of a solid substance from a liquid is called filter medium. Filter paper is the best example of a filter medium.

**(10) Filter:**

Any water insoluble porous material which has a reasonable degree of rigidity and can be used to separate the components from each other is called filter. Filter paper is a best filter.

**(11) Filtrate:** **(Rwp. 2012)**

That liquid which is collected after passing through the filter medium is called filtrate. It is collected in a beaker or flask in the laboratory.

**(12) Fluted filter paper:**

That filter paper which has a fan-like arrangement with alternate elevations and depressions at various folds, and is used to increase the rate of filtration is called fluted filter paper.

**(13) Gooch crucible:** **(Lahore 2012)**

A crucible which is made up of ceramic material having a perforated base and covered with a filter paper or asbestos material is known as Gooch crucible.

**(14) Mobile phase:**

A phase which is consisted of a single solvent or a mixture of solvents and is used for the separation of components in chromatography is called mobile phase.

**(15) Mother liquor:**

The solution which remains behind after the formation of the crystals is called mother liquor. Molasses from sugar industry is a best example of mother liquor.

**(16) Partition chromatography:** **(Lahore 2011, Rwp. 2013, Multan 2014, Guj. 2014, B. Pur 2014)**

That type of chromatography which involves the partitioning of the components between the two liquids.

**(17) Partition coefficient:** **(Sarg. 2014, B. Pur 2014)**

The ratio of the amounts of the solute dissolved in two immiscible liquids at equilibrium position is called partition coefficient. It is same as distribution coefficient and is represented by $K_D$.

**(18) Quantitative analysis:** **(Multan 2011)**

That branch of analysis in which the components of the sample are analysed quantitatively is called quantitative analysis. Volumetric and grammetric analysis are two major types of it.

**(19) Residue:**

That solid substance which is left behind on the filter medium during filtration is called residue. It cannot pass through the pores of filter paper.

**(20) Sintered glass crucible:** **(Lahore 2012)**

It is a glass crucible with a sintered glass disc sealed into the bottom.

**(21) Solvent extraction:**

It is a method of extracting of a desired component from the solutions by shaking it with second liquid in which the component is more soluble. This second liquid is immiscible with the first liquid. Separating funnel may be used to separate two layers.

**(22) Stationary phase:**

A stationary phase may be consisted of solid or a liquid supported on a solid or a gel. It may be packed in a column. In column chromatograph, silver gel is stationary phase.

**(23) Sublimand:** **(Lahore 2014)**

The solid substance which is being sublimed is called sublimand. Naphthalene can be sublimed and is called sublimand.

(24) **Sublimate:** (Lahore 2014)

That pure solid substance which is obtained after sublimation is called sublimate.

(25) **Sublimation:**

(Lahore 2009, Multan 2009, B. Pure 2013, Lahore 2014, Lahore 2014, Sarg. 2014, Rwp. 2014, D.G. Khan 2014)

The process of vaporization of a solid directly on heating without passing through the liquid phase and the condensation of these vapours on cooling to be solid, without passing through the liquid phase, is called sublimation.

(26) **Vacuum desiccator:**

A desiccator which is connected to the vacuum pump is called vacuum desiccator. It creates low pressure and causes water vapours to leave the substance and make it dry.

## ANSWERS TO THE SHORT QUESTIONS

### Purification Techniques

**Q.1 Mention various experimental techniques which are used for the purification of substances?** (Lahore 2007)

-1 کسی چیز کو صاف کرنے مختلف تجرباتی ہنروں کا ذکر کریں۔

**Ans:** The techniques (اصولی طریقہ کار) are as follows:

(i) Filtration (ii) Crystallization (iii) Sublimation (ٹھوس کی بخارات میں تبدیلی، عمل تسعید) (iv) Solvent extraction.

It depends upon (منحصر ہے) the nature of the substances, that which technique is to be used.

**Q.2 How do you justify that qualitative and quantitative analysis are discussed in analytical chemistry? (Multan 2011)**

-2 آپ کس طرح ثابت کریں گے کہ صفاتی اور مقداری تجزیہ کو تجزیاتی کیمیا میں زیر بحث لایا جاتا ہے۔

**Ans:** It is that branch of chemistry which gives up analysis (تجزیہ) of elements and compounds. In qualitative analysis (صفاتی تجزیہ), we come to know about nature of the elements and in quantitative analysis (مقداری تجزیہ) gives us the quantities of different elements in the compound.

**Q.3 Define sublimation with an example? (Rwp. 2005, Lhr. 2008, Multan 2008, (Sarg. 2009, Lahore 2009, Multan 2009, Rwp. 2009, Rwp. 2010, Bahawalpur 2011, D. G. Khan 2012, F. Abad 2012, Lahore 2013, Guj. 2013, Lahore 2014)**

-3 عمل تسعید کی تعریف کریں اور ایک مثال دیں۔

**Ans:** The vapourisation (بخارات میں تبدیلی کا عمل) of a solid directly on heating without passing through the liquid phase and the condensation of these vapours on cooling to solid without passing through liquid phase is called sublimation. Naphethalene, iodine, $NH_4Cl$, benzoic acid and camphor (کافور) undergo sublimation. We can do the separation of solids without using solvents.

**Q.4 How desiccator is used to dry the crystals? (Multan 2008, B.Pur 2009, Lahore 2012, Rwp. 2014, Sahiwal 2014)**

-4 قلموں کو خشک کرنے کے لیے ڈیسی کیٹر کیسے استعمال کیا جاتا ہے؟

**Ans:** The prepared crystals are wet and they need drying. In a desiccator (خشک کرنے والا), the crystals are spread on the watch glass and placed in a desiccator for several hours. Some drying agents (خشک کرنے والے) like anhydrous $CaCl_2$, silica gel or $P_2O_5$ are used.

**Q.5 How does a Gooch crucible increase the rate of filtration? (Gujranwala Board 2005, Multan 2007, Sarg. 2009, Rwp. 2011, Multan 2012, Sarg. 2014, Sahiwal 2014)**

-5 گوچ کی کٹھالی کس طرح فلٹریشن کی رفتار کو بڑھاتی ہے؟

**Ans:** This crucible is made up of porcelain (مٹی کا بنا ہوا). It has perforated base (پیندے میں سوراخ ہوں) covered with a filter paper or asbestos mate. Filtration can be done quickly if this crucible is placed in a suction (ہوا کا کھینچنا) of filtering apparatus.

**Q.6 Concentrated HCl and $KMnO_4$ solutions cannot be filtered by Gooch crucible. Give reason. (Gujranwala 2011, Bahawalpur 2011, F. Abad 2012, B. Pure 2013)**

-6 زیادہ ارتکاز والے HCl اور $KMnO_4$ کے سولیوشنز گوچ کٹھالی سے فلٹر نہیں ہو سکتے وجہ بتائیں۔

**Ans:** Conc. HCl and the oxidizing agents like $KMnO_4$ react with filter paper. For this purpose, the perforation (سوراخ) of Gooch crucible is covered with asbestos mate. In this way, the above solutions can be filtered.

## Crystallization

**Q.7 Which solvents are mostly used in crystallization. (Model Paper-2006-07, Rawalpindi 2007, Faisalabad 2007, Rwp 2011, Guj. 2013)**

-7 قلم کاری کے لیے کون سے سالونٹ مستعمل ہیں؟

**Ans:** The most commonly (کثرت سے ہونے والے) used solvents are:

(i) Water (ii) Rectified spirit (iii) Absolute alcohol (iv) Ether
(v) Acetone (vi) Chloroform (vii) $CCl_4$ (viii) Acetic acid (ix) Petroleum ether.

**Q.8 Give the main characteristics of the solvent used for crystallization.**
**(Sargodha 2005, Federal-2006, Sargodha 2008, Lahore 2009, Fd.Abad 2009, Guj. 2010, Faisalabad 2010, B. pure 2012, D. G. Khan 2012, Guj 2012. M. Pure 2012, D.G. Khan 2013, Lahore 2014)**

-8 قلموں کے بنانے میں کسی سالونٹ کی کیا صفات ہونی چاہئیں۔

**Ans:**
(i) The solvent should dissolve a large amount of solute at high temperature.
(ii) The solvent should have no chemical reaction with solute.
(iii) It should not dissolve the impurities (کثافتیں).
(iv) It should be cheap (سستا).
(v) It should not be inflammable (جلدی سے آگ نہ پکڑنے والا).

**Q.9 Mention the major steps involved in the crystallization.**
**(Model Paper-2006-07, Rwp-2007, Multan 2007, Lahore 2007, Sargodha 2008, 2011, Faisalabad 2013, F. Abad 2014)**

-9 قلمیں بنانے میں کونسے اہم اقدام کارفرما ہوتے ہیں؟

**Ans:** (i) Preparing the saturated solution (سیر شدہ سولیوشن). (ii) Filtering the impurities.
(iii) Cooling of the filtrate (فلٹر کیا ہوا مائع). (iv) Collection of crystals.
(v) Drying of crystals.

**Q.10 How the decolourization of undesirable colours and dehydration is carried out for freshly prepared crystalline substances? (Guj. 2008, B.P. 2008)**
**(Lhr-2006, Multan-2006, Rawalpindi 2007, Multan 2007, B.Pur 2007, Rwp. 2010, Federal 2013, D.G. Khan 2014, B. Pur 2014, Multan 2014)**

-10 تازہ تیار شدہ قلمی میٹریل کو بے رنگ کرنے اور پانی سے پاک کرنے کے لئے کیا کرنا پڑتا ہے؟

**Ans:** The decolorization (رنگ اُڑانا) of undesirable colours (نامناسب رنگ) is carried out by boiling the substance with sufficient amount of powdered animal charcoal in the solvent. Hot solution is filtered. In this way charcoal absorbs the coloured impurities and the pure decolorized (اُڑے ہوئے رنگ والے) substance crystallizes on cooling. The dehydrating agents employed are conc. $H_2SO_4$, $P_2O_5$ or silica gel.

## Solvent Extraction

**Q.11 What is solvent extraction? (Lahore 2008, Lahore 2010, Sarg. 2011, Mirpur 2012, Guj. 2013)**

-11 سالونٹ کی مدد سے سولیوٹ کو کیسے باہر نکالا جاتا ہے؟

**Ans:** This is a technique (طریقہ کار) in which a solute is separated (الگ کرنا) from the solution. For this purpose, the solution is shaken (ہلانا) with another solvent in which the solute is more soluble. Anyhow, the added solvent should not be miscible with the solution.

**Q.12 What is ether extraction?**

-12 ایتھر کی مدد سے کیمیائی مرکبات کو سالونٹ سے کیسے نکالا جاتا ہے؟

**Ans:** Some organic products are prepared in aqueous medium. We have to remove this organic compound from water. For this purpose, ether is added. Two separate layers are produced (دو الگ تہیں بنتی ہیں) in the separating funnel. The ether layer contains the organic compound. This ether layer is separated and ether is evaporated (بخارات میں تبدیل ہونا).

## Chromatography

**Q.13 What is $R_f$ value? Give its units.**
**(Multan Board 2005, D.G. Khan-2006, Multan 2007, Lahore 2009, B.Pur 2009, Faisalabad 2010, Faisalabad 2011, Multan 2011, Lahore 2012, Guj. 2013, Multan 2013)**

-13 $R_f$ کی قیمت کیا ظاہر کرتی ہے؟ اس کی یونٹس بتائیں۔

**Ans:** $R_f$ stands for (نمائندگی کرنا) retardation factor (ایسا فیکٹر جو رکاوٹ کو ماپے). It is different for each component which is separated by chromatography.

$$R_f = \frac{\text{Distance travelled by a component from the original spot}}{\text{Distance travelled by the solvent from the original spot}}$$

It has no units.

**Q.14 Give the main uses of paper chromatography.**
**(B.Pur-2006, Guj. 2009, Lahore 2010, B.Pur 2012, Multan 2013, Lahore 2014, Lahore 2014, D.G. Khan 2014)**

-14 پیپر کروميٹوگرافی کے اہم استعمالات بتائیں۔

**Ans:**
(i) For the separation and purification (صاف کرنے کا عمل) of coloured organic compounds.
(ii) For checking the purity (صفائی) of the compounds.
(iii) In qualitative and quantitative analysis.
(iv) For the separation, purification and identification (پہچاننا) of products of reactions.

**Q.15 Differentiate between stationary and mobile phase?**
**(Lahore Board 2005, Mirpur-2006, Federal-2006, Multan 2009, Faisalabad 2013, Guj. 2014)**

-15 حرکت پذیر اور ساکن فیز میں کیسے فرق کریں گے؟

**Ans:** The solvent or the mixture of solvents used for the separation of components in chromatography is called mobile phase (حرکت میں رہنے والا فیز). The phase over which mobile phase flows is stationary (ساکن) one. Water, ethyl alcohol etc. are some important mobile phases while silica gel and filter paper are some important stationary phases.

**Q.16 What is the distribution coefficient? To which technique it is applicable?**
**(Azad Jammu & Kashmir Board 2005, Guj. 2009, Lahore. 2010, D.G. Khan 2011, Gujranwal 2011, Guj 2012, D.G. Khan 2013, Multan 2013, B. Pur 2014, Guj. 2014, Guj. 2014)**

-16 سولیوٹ کا سالیوشن میں تقسیم کا مستقلہ کیا ہوتا ہے؟ یہ کس ہنر مندی میں استعمال ہوتا ہے؟

**Ans:** It is the ratio of the amounts of solute dissolved in the immiscible liquids (مائعات جو حل نہ ہو سکیں) at equilibrium.

$$\text{Distribution coefficient } (K_D) = \frac{\text{concentration of solute in organic phase}}{\text{concentration of solute aqueous phase}}$$

This technique is used in the solvent extraction of certain soluble compounds.

**Q.17 How naphthalene can be purified?** **(Guj. 2008)**

-17 نیفتھالین کو کس طرح صاف کیا جا سکتا ہے؟

**Ans:** Naphthalene can be directly converted from solid to vapour state, i.e., by the sublimation. The impurities are left behind (کثافتیں پیچھے جاتی ہیں) because they do not sublime. Inverted funnel can be used.

**Q.18 In solvent extraction technique, repeated extractions using small portions of solvent are more efficient than using a single extraction but larger volume of solvent. Why? (Rwp. 2008, Sarg. 2010, Lahore 2011, Sarg. 2014)**

-18 جب ایک نامیاتی سالونٹ سے ہم کسی سولیوٹ کو دوسرے سالونٹ کی مدد سے الگ کرتے ہیں تو نامیاتی سالونٹ کے چھوٹے چھوٹے حصے لے کر کام کرنے سے سولیوٹ زیادہ نکالا جاسکتا ہے بجائے کہ سارا نامیاتی سالونٹ ایک ہی مرتبہ استعمال کر لیا جائے۔ کیسے؟

**Ans:** It is due to constant value of distribution coefficient (تقسیم کا عددی سر) of a solute in two solvents, which are insoluble. Say an organic solute is 1g, and is present in 1000 ml of $H_2O$. We have 1000 mL of ether for extraction. If we use 1000 mL ether in one installment, 67% of 1g of solute is extracted if $K_D$ is 2 in the favour of ether. But by using two installments of 500 mL each 75% of 1 g is extracted and so on.

**Q.19 Why is there a need to crystallize a crude product? (Guj. 2010, Lahore 2014)**

-19 ناخالص پراڈکٹ کو صاف کرنے کی کیا ضرورت ہے؟

**Ans:** Impurities are mostly present in the crude product (ناخالص پراڈکٹ). By using a suitable solvent, in which the required substance under preparation is soluble is used to get the crystals of that substance. The crystals are a pure substance.

**Q.20 Iodine is more soluble in water in presence of KI. Give reason.**

**(Faisalabad 2011)**

-20 $I_2$ کو پانی میں حل پذیری کے لئے KI کی ضرورت ہوتی ہے۔ سبب بتائیں۔

**Ans:** To dissolve $I_2$ in water a dilute solution KI should be prepared first of all. $I_2$ combines with $I^{\ominus}$ of KI to give $I_3^{\ominus}$ ion. This ion $I_3^{\ominus}$ is soluble in water. $I_2$ is not soluble in $H_2O$.