



B.Sc. Third Year V Semester ANALYTICAL CHEMISTRY

Paper XIII [Applied Analytical Chemistry-I]



Analysis of Cosmetics

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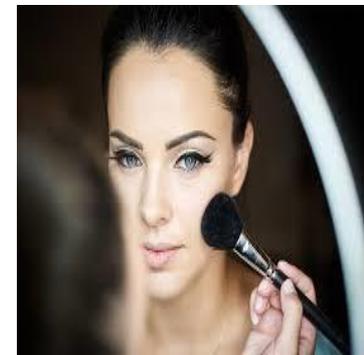
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Analysis of Cosmetics

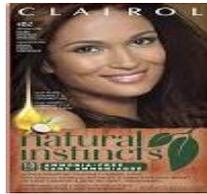


Introduction

- ❑ Word **Cosmetic** is originated from Greek word **kosmeticos** means *adorn* (Make more beautiful or attractive) and *preparation* (to improve person's appearance)
- ❑ **Cosmetics** are substances or products used to enhance or alter the appearance of the face or fragrance and texture of the body. Many cosmetics are designed for use of applying to the face, hair and body. They are generally mixtures of chemical compounds, some being derived from natural sources and some being synthetics or artificial.

Defination

- ❑ It is external preparation meant for applying on external parts of the body i.e. nails, skin, hairs, for coloring, covering, softening, cleaning, nourishing, waving, setting, preservation, removal and protection.
- ❑ Other way of defining cosmetic is, **It is item intended to be rubbed, poured, sprinkled, introduced in to, otherwise applied to the human body or part thereof cleaning, beautifying, promoting attractiveness or altering the appearance.**



- ❑ **Common Make-up Items Include:** lipstick, mascara, eye shadow, foundation, whereas other common cosmetics can include skin cleansers and body lotion, shampoo and conditioner hair styling products perfume and **cologne**.
- ❑ Ex. Nivea, L'Oreal, Olay, Dove, Ponds, **Fair & lovely, Kajal, Kumkum, Lipstick, Hair dyes.**



Good Characteristics of Cosmetics

❑ Cosmetics are prepared by a single or mixture of two component to intended required action but such articles are not included like soaps.

❑ The sensory experience of cosmetics product is based on its appearance, texture and odor, and is measure of its quality. Customer acceptance of cosmetic product is largely based on physical appearance of product mainly feels to the touch.

1. A smooth & shiny appearance • Should make face pleasant to look and touch.

2. Freedom from sweating • Must adhere to skin. • Must have pleasant smell.

3. A suitable degree firmness during reasonable variations of climatic temperature.

4. Retain plasticity without any tendency to dry out or crumble (good covering power).

5. Adhesiveness, how will cling to the face & absorbency.

6. Excellence impairing bloom, colour and good absorbency water.

7. Preservatives & other additives.

8. Staining, skin with staining dyes (like lipsticks and other creams).

9. Elevating transitions to skin due to heat.

10. Physical appearance colour dispersions & controlled.

11. Disintegration tissue.

12. Hardness.

13. Melting range.

14. Softening point.

• Must produce a lasting effect , so that frequent application is unnecessary.

• Also provide good slip, cooling effect & efficient lubrication .



Classification of Cosmetics

□ Depending upon physical form of cosmetics are classified as:

Types of Cosmetics



Other Cosmetics

- Face powder, Compact, Body powders
- Lipsticks
- Sunscreen, Massage cream
- Many cure & Paddy cure



Classification of Cosmetics



□ According to their cosmetic functions mainly divided into four types.

1. **Decorative cosmetics:** A preparation designed to beautify and improve the complexion, skin, hair etc. or substance used to enhance appearance or odor of the human body.

Ex. Nail enamels, Hair **propins**, **Lipsticks**.



2. **Corrective cosmetics:** Face powders, Rouges.

3. **Protective cosmetics:** Creams, Heavy face powders

4. **Curative cosmetics:** Anti perspirants, **Deodorants**.



□ According to their action or use on different part cosmetics also divided as:

□ Face cosmetics: Powders

□ Skin cosmetics: Powders, creams and Lotions.

□ Hair cosmetics: Hair dyes.

□ Nail cosmetics: Nail enamels and removals.

□ Eye cosmetics; Eye shadows, Kajal. **Powders**



□ **Powder**, is a fine, loose, dry newly fallen snow and dry particles produced by grinding, crushing of solid substances.

□ **Four types:** a) Face powders b) Compact Face powders c) Talcum powders d) Baby Powders

□ Face Powders are loose face powder and compact face powder.

□ Face powder is a basically a cosmetic product which its function is the compliment skin colour, it should enhance the appearance of the skin by making a shine due to secretion of sebaceous and sweet glands.

BEST TALCUM POWDER



❑ **Talcum powder:** is a white loose substances (powders) are used all over your body, mostly after a bath or shower to reduce moisture.

Amongst the various cosmetics, body powder is one of the widely consumed cosmetic preparations.

Main use of body powders is to **absorb moisture** or **perspiration** specially after bathing particularly in warmer countries. Colour is not used in body powders.



❑ **Talcum powder** is one of the most popular beauty care product used by men and women including infant to keep the skin dry, to suppress the bad (sweat) odor and to feel fresh. Three types of talcum powders are generally found in the market – for men, for women and baby powder.

❑ Ex. Spinz, Ponds, Jonson & Jonson, Liril, **Cinthol**, Dermi cool.



❑ All talcum powder is enriched with nice fragrances which make each and every end user to stay active throughout his/her working day, or during travel or any other occasion. Talcum powder helps in giving a real glow to skin and relieves skin from nice fragrance and keeps entire day active and fresh. It gives relief to irritated skin and prevents chafing. Talcum powder is used on an infant to keep skin softer and keeping them cheerful and happier.

❑ **Face powder:** is loose powder used for same purpose in addition to covering out your skin tone but on your face only.

• Face powder is an indispensable (absolutely necessary) article of a lady's cosmetic range.

❑ Ex. Lakme, Cinique, Loreal, Matte.

❑ **Compact** (also powder box and powder case) is a cosmetic product. It is usually a small round metal case and contains two or more of the following: a mirror, pressed or loose face powder with a gauze sifter and a powder puff. Used to give skin smooth complexion.

• A face powder is basically a cosmetic product which has as its prime function the ability to complement skin colour by imparting a velvet finish to it.



Key Ingredients

Ingredient	Category	Use
Talc	Base/Mineral	Naturally occurring mineral added in safe amount.
Mica	Covering Agent	It helps the powder to stay on the skin longer.
Kaolin	Slip/Absorbent	Enhance coverage while reducing the sheen provided by talc.
Magnesium Stearate	Adhesive	Improves the consistency of face powders, ensuring a smooth application
Calcium Carbonate	Absorbent	Absorbs moisture to minimize oiliness and create a matte finish.
Inorganic & Organic Pigments	Colorant	Added to the formula to create the desired tint.
Triclosan	Perfumes	Enhance the intrinsic appeal.

Features:

- Accurately processed Skin-friendly
- Effective usage
- Free from harmful chemicals
- Hygienically processed Long shelf life

General Composition of Talcum powder or Face powder

- ❑ Talc: Natural starch potato rice.
- ❑ Bases: China clays (Kaolin)
- ❑ Chalk: Calcium carbonate.
- ❑ Magnesium carbonate.
- ❑ Covering pigment: ZnO, TiO₂.
- ❑ Adhesive: Zn, Mg streates.
- ❑ Boric Acid.
- ❑ Bismuth chloride and nitrate.
- ❑ Perfumes

Percentage Composition of Talcum powder or Face powder

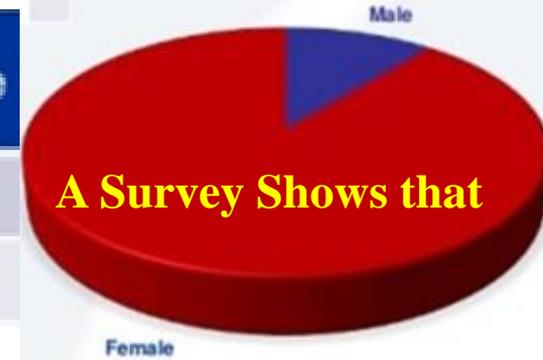
- Talc  7.0 gm
- Kaolin  A Pinch
- Silica
- Starch
- Calcium Carbonate 0.5 gm
- Zinc Oxide 0.25 gm
- Zinc stearate 0.5 gm
- Boric acid 0.3 gm
- Perfume 1- 2 Drops

Depending on Nature of Skin

Type of Skin	Type of Powder	Characteristics
Dry	Light	<ul style="list-style-type: none"> • Slight Covering Power • Contain large Quantity of Talc
Normal or Moderately Oily	Medium	<ul style="list-style-type: none"> • Comparatively higher covering power • Lesser talc and balanced by zinc oxide
Very Oily	Heavy	<ul style="list-style-type: none"> • High Covering Power • Lower quantity of talc and High quantity of zinc oxide.

Category	Percentage
Male	11
Female	89

%USED BY



Analysis of Face Powder or Talcum Powder

- ❑ Face powders or Talcum powders are generally analyzed for their density, viscosity, moisture content, ash content, heavy metals like zinc, aluminium, magnesium and boric acid.
- ❑ **Preparation Sample Solution:** Weigh accurately 2-3 grams of face powder or talcum powder sample into a 250ml beaker. Add to it 5ml of conc. HCl, and add necessary amount of distilled water in it. Heat the contents to boiling, cool it, filter through a filter paper into a 250ml standard flask. Thoroughly wash the beaker and filter paper with hot distilled water, cool the flask and contents to maintain the room temperature. Dilute the volume with distilled water up to the mark, shake and mix well.

□ These involves conversion of zinc and aluminium into their hydrochlorides reactions are as,



□ This is a sample solution contain Al, Zn, Mg and Boric acid contents and used as a sample solution for determination of Zinc, Aluminium, Magnesium and Boric acid in powders.

Procedures:

□ **Determination of Zn & Al:** Take the suitable aliquot of the above sample solution which contains 10-20mg of Al and 20-40mg of Zn approximately. Add 1-2 drops of phenolphthalein indicator, then 2N of ammonium hydroxide solution till solution become slightly turbid. Add 5ml CH_3COOH , dilute the content up to 100ml with distilled water. Heat it 70-90 °C for some time, then add 10mg 8-hydroxy quinoline, add slowly aluminium acetate solution into filtrate until PH of solution becomes 4.8-5.

□ Heat it again for 2-3 minutes below boiling point, kept at side, the residue filter through a sintered glass crucible. Wash thoroughly with water. Dry it 1-2 hours at 130-140°C in an oven, cool and weigh till content weight of residue is obtained.

□ **Calculations:**

$$\% \text{ of Zn} = \frac{\text{wt. of Zn ppt} \times 0.1848 \text{ factor}}{\text{wt. of sample taken}} \times 100$$

$$\% \text{ of Al} = \frac{\text{wt. of Al ppt} \times 0.0587 \text{ factor}}{\text{wt. of sample taken}} \times 100$$

□ **Determination of Mg:** Take 10ml of above talcum powder aliquot of sample solution on a conical flask. Add to it 5ml buffer pH¹⁰ solution, then add 4-5 drops indicator in it. Titrate these solution with standard 0.1M EDTA solution from burette, till colour changes wine red to blue.

□ **Calculations:**

$$1\text{ml of } 0.1\text{M EDTA} = 0.007268\text{grams Mg content}$$

❑ **Determination of Boric Acid:** By Ion Exchange Chromatography Method

❑ A glass column of 50cm long and 2cm diameter is taken. A glass wool is inserted at the bottom of a column, fill column tube with water, add to it amberlite IR 120 ion exchange resin slowly up to 20cm column length. Wash with HCl and then fill the remaining with water. Place face powder sample solution containing approximately 100-200mg of boric acid in a beaker. Add 2 drops of phenolphthalein and 10% NaOH solution make it with alkaline. Evaporate to dryness on steam bath, dry the residue in an oven at 140°C for one hour. Cool it at temperature, add to it 50ml hot water. Acidify with HCl and filter hot solution through ash less filter paper, collect filtrate, repeat the procedure with residue till nearly 200ml filtrate is collected.

❑ Pass this solution through ion exchange column till 10 to 15 minute, elute 15ml water and collect elute. Make it alkaline by NaOH and then acidify with HCl, reflux for 5 minutes. Again neutralize with NaOH and 4 to 5grams of manitol and 1-2 drops of phenolphthalein. Titrate these with standard 0.1N NaOH solution till pink colour appears. Perform blank titration, calculate boric acid as.

❑ **Calculations:**

Viscosity:

1ml of 0.1N NaOH = 0.00615grams of Boric acid

- The **viscosity** of a fluid is a measure of its resistance to gradual deformation by shear stress or tensile stress. For liquids, it corresponds to the informal concept of "thickness": for example, syrup has a higher **viscosity** than water.
- Units for viscosity are Poise.
- Viscosity measurements are made using various Viscometers.

Density:

- Density is defined as its mass per unit volume and is measured in kg/m^3 . The symbol most often used for density is ρ .
- Densities of samples are determined by dividing their mass to the volume they occupy.

Hair Dyes

Meaning of Hair Dye:

□ Hair dye or hair coloring is the practice of changing the hair color. The main reason for this are cosmetic to cover gray hairs or to change the color of hair which is regarded as more fashionable or desirable in today's days world. **History of Hair Colors:**



□ Early **Egyptians** used for the first hair color.
□ First culture to colour hair.

□ Used: **Henna** – Red hues, **Chamomiles** – Blonde Hues, **Sage** – Brown Hues.

□ Red hair was popular due to **Queen Elizabeth's red hair**.
□ Women would apply mixture of black sulfur, alum and honey and sit in the sun until the hair has turned a golden.

□ In 1907, **Eugene Schueller**, a young **French chemist** developed an innovative hair-colour formula. He called his improved hair dye *Aureole*. Schueller formulated and manufactured his own products, which he then sold to Parisian hairdressers, using the chemical **Paraphenylenediamine (PPD)**.

□ **Early 20th Century**

□ Popular for actresses to color their hair.

□ It wasn't until the middle of the century that a common woman dying her hair was accepted.



Eugene Schueller

- Advantages of henna:
 - Non-toxic,
 - Non sensitizing to skin
- But laborious work to prepare henna paste
- Following composition is generally used nowadays:

Powdered henna	89%
Pyrogallic acid	6%
CuSO ₄	5%
- Concentration of pyrogallic acid responsible for color

What is hair dye?

- ❑ **Hair Dye or Hair Color:** is a chemical tool that is used to change the color of a person's hair. Used mostly to change gray hair a sign of an advanced age.
- ❑ Younger people that used hair dye use it as a fashion.

About our Hair:

- ❑ Human hair is made of **keratin, water and dead cells.**
- ❑ Each strand of hair is made up of three layers, **Cuticle, Cortex & Medulla.**

Marketed products

- Godrej,
- Garnier,
- L'Oreal,
- Lakme,
- Indica,



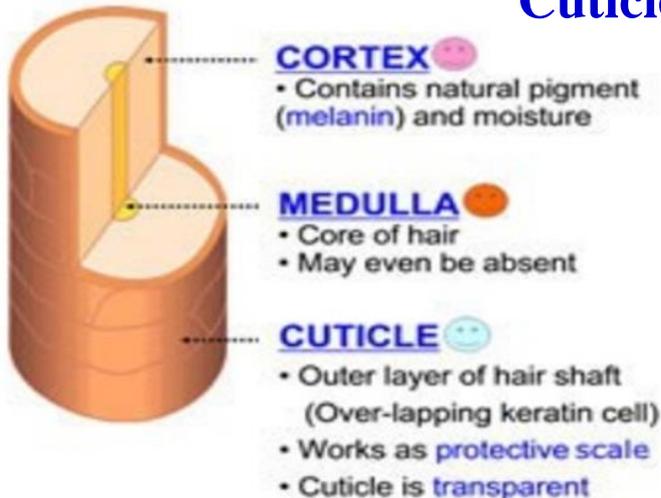
Cuticle: It is the outermost layer of the hair and consists of keratinized. It is made up of scale like protein cells which are overlapping. Protein cells protect the cortex. Natural black hair color is due to the melanin clusters of dispersed within the colorless keratin based cortex of hair.

Cortex:

It is located peripheral to the medulla and forms then major part of the shaft. It consists of elongated cells, containing pigment granules in dark hair while air in white hair.

Medulla:

It is the central part of the shaft and is generally noticeable in thick hairs. It is composed of two or three rows of polyhedral cells containing pigment granules and air spaces.



CORTEX (pink circle)
• Contains natural pigment (melanin) and moisture

MEDULLA (orange circle)
• Core of hair
• May even be absent

CUTICLE (blue circle)
• Outer layer of hair shaft (Over-lapping keratin cell)
• Works as protective scale
• Cuticle is transparent



Reason for Hair Color:

- To change the natural color of the hair.
- To color the gray hair.
- To change the color of the hair temporarily for a particular occasion.



Ideal Characteristics of Hair Colors:

- Color hair without reducing natural gloss.
- Not change the texture of hairs. **Not have systemic toxicity.**
- Not injure hair shaft. **Non-irritant & free from sensitization.**
- Color of dyed hair stable to physical factors. **Stable in the formulation sold in market.**
- No effect of shampoos, brilliantness, setting lotion on the dyed hair.

Types of Hair Colors:

- Temporary hair color.
- Semi permanent hair color.
- Permanent hair color.



Temporary Hair Colors:

- Temporary hair color is available in various product forms including rinses, shampoos, gels, sprays, foams. This type of hair colors is typically used to give brighter, more vibrant shades or colors such as orange or red, that may be different to achieve with semi-permanent and permanent hair color. While temporary hair color products hold a lesser market than semi-permanent and permanent agents., they have value in that they can be easily and quickly removed without bleaching or application of a different coloring product. These colorants donot penetrate into cortex or medulla. As such the dye is easily removed with shampoo so known as *temporary hair color*.

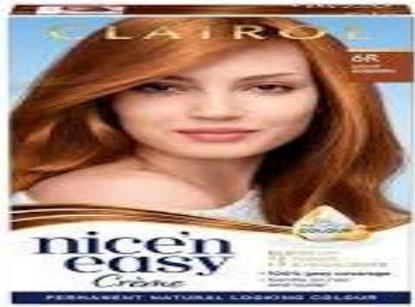
Semi-Permanent Hair Colors:

- ❑ Differ from color shampoo with respect to time.
- ❑ Give stronger & more permanent coloration to hair.
- ❑ Some colors are removed in 4-8 shampoos.
- ❑ Dyes used are Nitrophenylenediamine, Nitroamineophenols, Mixture is prepared before preparing color shades.
- ❑ Should be studied on white wool or hair. Semi hair color has no Ammonia.



Permanent Hair Colors:

- ❑ All 'permanent' hair color products and lighteners contain both a developer or oxidizing agent, and an alkalizing ingredients as part of their ammonia or an ammonia substitute.
- ❑ When the tint (color) containing the alkalizing ingredients is combined with developer (usually hydrogen peroxide), the peroxide becomes alkaline and diffuses through the hair fiber, entering the cortex, where the melanin is located.
- ❑ The lightening occurs when the alkaline peroxide breaks up the melanin and replaces it with new color.

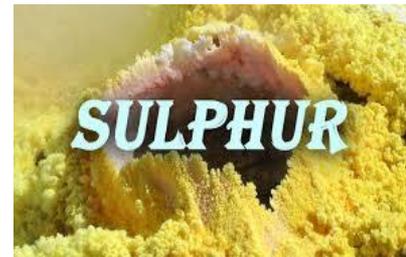


General Composition of Hair Dye

- ❑ Hair dyes contains pigments substances which are part of hair cosmetics.
- ❑ They are present in the form of an aqueous solution and contains different forms of dyes for coloring purposes of hair.
- ❑ Hair coloring is the process of change in the hair colors.
- ❑ **Hair dye contains:** Para phenylene diamine (PPD).



- : Lead salt in the form of lead acetate
- : Glycerol and glycol
- : Sulphur in the form sulphate
- : Waxes
- : Fatty materials
- : Hydrogen peroxide
- : Ammonia
- : Perfumes
- : De-ionized water



Examples



- : Henna dye
- : Godrej hair dye
- : Garnier hair dye
- : L'Oreal hair dye
- : Lakme hair dye
- : Indica hair dye

How to Choose Henna Hair Dye from NMP Henna Powder



Analysis of Hair Dyes:

Procedures:



I] Determination of Non-volatile matter:

- ❑ Weigh accurately about 0.3gm of hair dyes in the weighing bottle or Petri dish.
- ❑ Heat it in an oven at 105°C for 2 hours.
- ❑ Cool it in a desiccator and weigh as non-volatile matter.
- ❑ Calculate the percentage of non-volatile matter by usual calculations

❑ Calculations:

$$\% \text{ of Non-volatile matter} = \frac{\text{Weight of sample after heat}}{\text{Weight of sample taken}} \times 100$$



II] Determination of Ash content:

- ❑ Weigh 1gm of hair dye in previously weighted platinum dish or crucible.
- ❑ Heat on a steam bath until hair dye solution boiled.
- ❑ Add 2gm of ash less cellulose powder and mix thoroughly with glass rod.
- ❑ Wipe the glass rod with ash less filter paper and add the same in to dish or crucible.
- ❑ Carefully charge the mixture on IR heating lamp.
- ❑ Place these charged mixture in Muffle furnace .
- ❑ Heat at 600°C till complete ashing of sample take place.
- ❑ Cool and weigh ash content.



❑ Calculations:

$$\% \text{ of Ash Content} = \frac{\text{Weight of sample after heat}}{\text{Weight of sample taken}} \times 100$$



III] Determination of Lead content:

- ❑ Shake the hair dyes sample well before use for analysis.
- ❑ Take 25ml of sample of hair dyes, into a 250ml beaker.
- ❑ Evaporate the content nearly to dryness on steam bath.
- ❑ Add 20ml of H_2SO_4 and HNO_3 in 1:1 proportion covers the precipitate content with watch glass.
- ❑ Heat the content to oxidize any organic matter after completion of oxidation remove the watch glass.
- ❑ Evaporate to completely remove SO_2 fumes nitrous fumes.
- ❑ Cool and dilute with 50ml distilled Water .
- ❑ Again heat the content up to boiling and stirred well.
- ❑ Add 50-70ml distilled water and allow to stand for one hour.
- ❑ Filter it through ash less filter paper.
- ❑ Wash it with dilute H_2SO_4 , cool, dry and ignited it.
- ❑ Residue obtained is the amount of lead sulphate ppt and weigh as lead sulphate as lead and calculate it by usual method.



❑ Calculations:

$$\% \text{ of Lead Content} = \frac{\text{Weight of lead sulphate ppt} \times \text{Factor } 0.6832}{\text{Weight of sample taken}} \times 100$$

IV] Determination of Sulphur content:

- ❑ Take 5ml of hair dye sample solution in centrifuge tube, centrifuged the content.
- ❑ Decant the aqueous part, again add 10ml of water again centrifuged it and decant it.
- ❑ Oxidize the residue with bromine water on steam bath.
- ❑ Sulphur in the residue is precipitated as barium sulphate by adding hot barium chloride solution.
- ❑ After ppt boil contents, cool it and filter through ash less filter paper.
- ❑ Wash, dry and ignite the ppt and weigh as barium sulphate.
- ❑ Calculate the percentage of sulphur by usual calculation.

❑ Calculations:

$$\% \text{ of Sulphur Content} = \frac{\text{Weight of barium sulphate ppt} \times \text{Factor } 0.1376}{\text{Weight of sample taken}} \times 100$$

V] Determination of Glycerol content:

- ❑ Place accurately 5gm weighed hair dye sample in to a separating funnel.
- ❑ Add 20ml water, mix the contents and make it slightly acidic by adding dil. HCl.
- ❑ Add 20ml methanol and 20ml chloroform and mix it, shake well and take out the layer separated.
- ❑ Drawn out lower layer, extract the aqueous layer with another 20ml CHCl_3 .
- ❑ Add 10ml of 2-methoxy ethanol to the aqueous extract and evaporate to 10ml.
- ❑ Transfer it to a centrifuge tube, immerse the lower portion of centrifuge tube in steam bath and vigorously boil.
- ❑ Evaporate till 4-5ml contents remain again add 3-5ml 2-methoxy ethanol and repeat

evaporation.

- ❑ Add an accurately measured volume of triethylene glycol solution to evaporated.
- ❑ Insert 5ml of prepared sample solution into a gas chromatography column, examine the curve obtained.
- ❑ Prepared standard solution containing known amount of triethylene glycol and examine the curve of gas chromatography extensive peak.
- ❑ Calculate the peak height ratio from the sample and known amount of glycerol.

❑ Calculations:

$$R_s = \frac{\text{Peak height of glycerol in sample}}{\text{Peak height of internal standard solution}}$$

$$R_{std} = \frac{\text{Peak height of glycerol in standard solution}}{\text{Peak height of internal standard solution}}$$

$$\% \text{ of glycerol content} = \frac{R_s \times \text{mg of glycerol in standard} \times \text{mg of internal standard sample}}{R_{std} \times \text{mg of internal standard solution}} \times 100$$

❑ Samples Preparation of PPD:

Proportional random sampling was used to select 15 henna salons from three cities in the UAE. Twenty five henna samples were bought from these selected salons, 11 of them were black henna and the remaining 14 were red.

❑ Analysis of Samples for PPD:

- ❑ PPD standard (0.11 mg per ml) was prepared by weighing pure PPD substance (0.011 gram) and dissolving it in 50 % aqueous methanol solution (100 ml).
- ❑ High-performance Liquid Chromatography (HPLC) was performed using an Agilent 1100 HPLC Series equipped with a Refractive Index Detector (RID) with a wavelength of 290 nm and a spectrum range of 190 to 400 nm.
- ❑ The HPLC conditions were: mobile phase, 0.05 M acetic acid-methanol (95/5) and adjusted to a pH of 5.9 with ammonia; the temperature was 30°C; flow rate was 1.5 ml/min; wave-length 290 nm; pressure 174 bar; and column.
- ❑ One gram of each of the collected samples was weighed into a 50 ml volumetric flask and diluted with 50% aqueous methanol solution (50 ml).
- ❑ This solution was then filtered after 15 minutes. Finally one ml of this solution was diluted to 5 ml with 50% aqueous methanol solution and analyzed for PPD.
- ❑ To confirm the identity of PPD in our samples, one ml of the standard was diluted to 5 ml with 50% aqueous methanol solution and analyzed before analyzing any sample to determine its spectrum and its retention time.

.....SUGGESTIONS ?

Created by, Dr. Subhash Lonkar

Thank you.....

