Benha university	**4^{th} year students**	**Date : 13.01.2019**
Faculty of science	**Adv. Analytical chemistry**	**Time: 2 hours**
Chemistry department	**(Spec. chemistry)**	**Code: Ch440**

Answer the following questions:

Q1: (20 Marks) Determine the color space (LCH) and the color of inorganic pigment using CIE-LCH method and the following data:

- **i. (10 Marks)** L= 60, a= 50 and b= 15
- **ii. (10 Marks)** L= 30, a= 12 and b= 45

Q2: (20 Marks)

A. (10 Marks) Mention the different types of ion selective electrode with explain only one with example.

B. (10 Marks) How can you clean the glass electrode?

Q3: (20 Marks)

A. (10 Marks) Discuss the basic components of diffuse reflectance spectroscopy and explain your answer with schematic diagram.

B. (10 Marks) Define color space and its types.

Q4: (20 Marks)

A. (10 Marks) Mention applications of ion selective electrode.

B. (10 Marks) X-rays of wavelength 1.5405 A are diffracted from crystal at an angle 36^o. Assuming that the distance between layers in the crystal (d) =2.44A, (hkl=311) and β= 0.85^o calculate the crystal size in nm. (1 nm = 10A).

Q5: (20 Marks)

A. (10 Marks) Mention the advantage and disadvantage of ion selective electrode

B. (10 Marks) Explain the accuracy and precision

***Best Wishes, Dr. Ayman Abdel Razik and Dr.* Ehab Abdelrahman**

1. The color space (LCH) and the color of inorganic pigment using CIE-LCH method and the following data:

iii. **(10 Marks)** L= 60, a= 50 and b= 15

iv. **(10 Marks)** L= 30, a= 12 and b= 45

Answer:

$C= (a^2+b^2)^{1/2}$ **$h=ten^{-1}$** (b/a)

L= 60, C=52, h= 73 ^{o} : color is light red

L= 30, c= 47, h =75 ^{o} : color is dark red

Mention the different types of ion selective electrode with explain only one with example.

1. Glass membrane (i.e. H+ electrode)

2. Solid-state electrode (e.g. F- electrode uses a Eu2+-doped LaF3 crystal)

3. Liquid-based electrode (e.g. Ca2+ electrode uses a liquid chelator)

4. Compound electrode (e.g. CO2 gas sensing electrode)

Solid-state electrode

This is based on an inorganic crystal doped with a small amount of ions of a different valency to create vacant sites.

For instance, in a F- ion electrode, a LaF3 crystal is doped with EuF2 so that there are anion vacancies for the migration of F through the LaF3 crystal, Response of F- ion electrode E = constant - (0.05916) log (AF-, outside) = constant - (0.05916) log gF- - (0.05916) log [F-]

At high pH, there is interference by OH- (Note kF-, OH- = 0.1)

At low pH, F- is converted to HF (pKa = 3.17) to which the electrode is insensitive.

How can you clean the glass electrode?

Often the pH meters are used in applications, which require regular cleaning of the electrode. These applications involve very hard waters, dirty samples like soil slurries, viscous materials or samples with high oil and protein content. We do not recommend these procedures for persons unfamiliar with or unable to use safe techniques involving these chemicals: Detergents, HCl (Hydrochloric Acid), and NaOH (Sodium Hydroxide).

Soak the electrode in a 0.4 molar concentration of HCl (hydrochloric acid) for 10 minutes, then rinse the electrode with deionized or distilled water. This should remove any organic protein from the glass electrode and the surface of the reference electrode.

Soak the electrode in a 3.8 or 4.0 molar KCl (potassium chloride) solution heated to 50°C for 1 hour. Allow the KCl solution to cool down to room temperature, then rinse the electrode with deionized or distilled water. This will open and clean the reference electrode of all contaminants.

Soak the electrode in a 4.01 pH buffer solution (EC-BU-4BT), heated to 50°C for 1 hour. Allow the buffer to cool down to room temperature, then rinse the electrode with deionized or distilled water. This will open and clean the reference electrode.

After each use, rinse the electrode in 0.5 N or 1% HCl. If you have a build-up of oil or protein contaminants, try soaking the electrode in warm detergent and water solution. Degreasing dishwashing detergents or stain removing pre wash pretreatment are ideal for this: any brand will do. An overnight soak may be needed if build-up is heavy. Then rinse the pH sensor in deionized or distilled water and soak for 10 minutes in 1% HCl. Rinse the pH sensor in deionized or distilled water and check in buffers. If the pH sensor calibrates to buffers it can be used in tests. When the pH electrode cannot be calibrated even after attempts to clean it, it must be replaced.

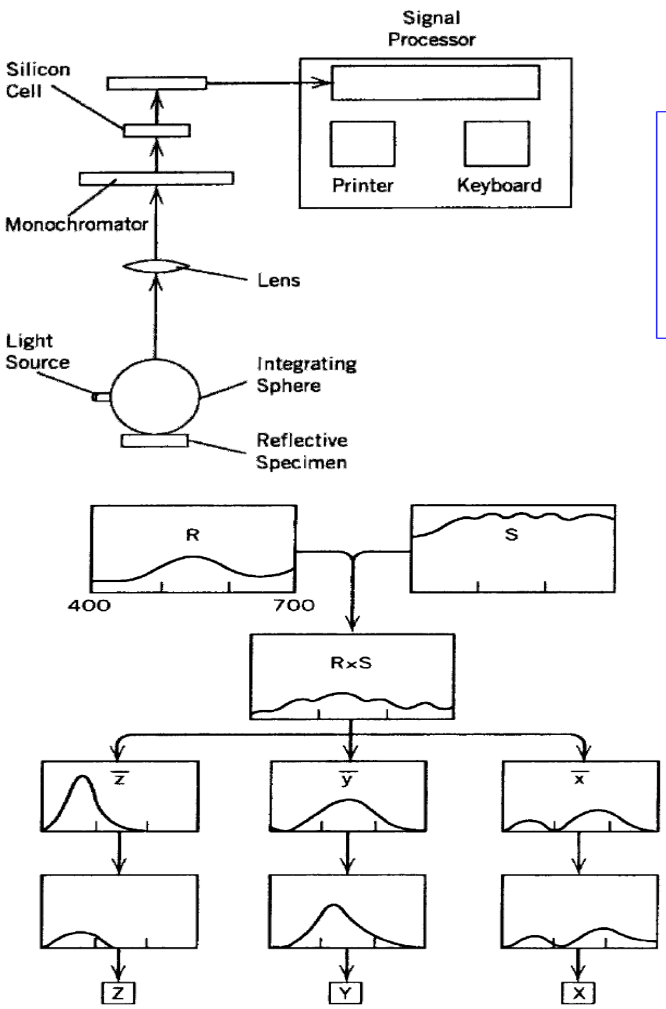
For protein removal, soak the pH electrode in contact lens enzymatic cleaner solution overnight. The enzymes will remove proteins from glass and plastic.

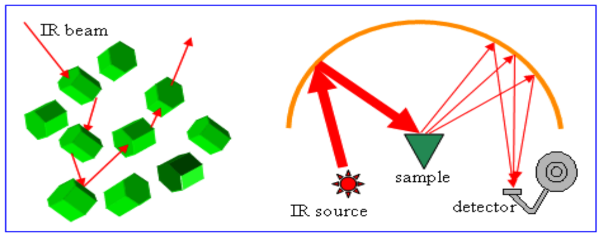
Discuss the basic components of diffuse reflectance spectroscopy and explain your answer with schematic diagram.

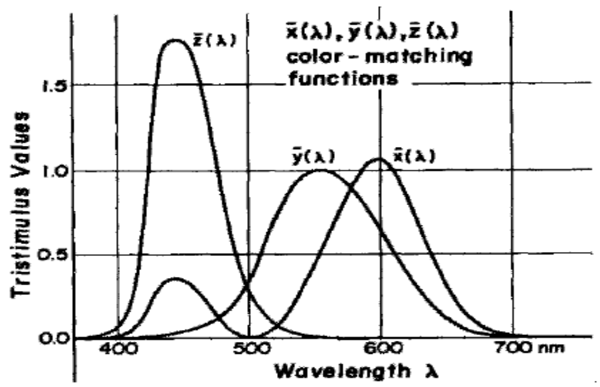
Spectroscopy is the study of light as a function of wavelength that has been emitted, reflected or scattered from a solid, liquid, or gas. Absorption and Scattering: As photons enter a mineral, some are reflected from grain surfaces, some pass through the grain, and some are absorbed. Those photons that are reflected from grain surfaces or refracted through a particle are said to be scattered. Scattered photons may encounter another grain or be scattered away from the surface so they may be detected and measured. Photons may also originate from a surface, a process called emission. All natural surfaces emit photons when they are above absolute zero. Emitted photons are subject to the same physical laws of reflection, refraction, and absorption to which incident photons are bound.

Photons are absorbed in minerals by several processes. The variety of absorption processes and their wavelength dependence allows us to derive information about the chemistry of a mineral from its reflected or emitted light. The human eye is a crude reflectance spectrometer: we can look at a surface and see color. Our eyes and brain are processing the wavelength-dependent scattering of visible-light photons to reveal something about what we are observing, like the red color of hematite or the green color of olivine. A modern spectrometer, however, can measure finer details over a broader wavelength range and with greater precision. Thus, a spectrometer can measure absorptions due to more processes than can be seen with the eye. Today, spectrometers are in use in the laboratory, in the field, in aircraft (looking both down at the Earth, and up into space), and on satellites. Reflectance and emittance spectroscopy of natural surfaces are sensitive to specific chemical bonds in materials, whether solid, liquid or gas. Spectroscopy has the advantage of being sensitive to both crystalline and amorphous materials, unlike some diagnostic methods, like X-ray diffraction. Spectroscopy's other main advantage is that it can be used up close (e.g. in the laboratory) to far away (e.g. to look down on the Earth, or up at other planets).

Spectroscopy's historical disadvantage is that it is too sensitive to small changes in the chemistry and/or structure of a material. The variations in material composition often cause the shift in the position and shape of absorption bands in the spectrum. Thus, with the vast variety of chemistry typically encountered in the real world, spectral signatures can be quite complex and sometimes unintelligible. However, that is now changing with increased knowledge of the natural variation in spectral features and the causes of the shifts. As a result, the previous disadvantage is turning into a huge advantage, allowing us to probe ever more detail about the chemistry of our natural environment.

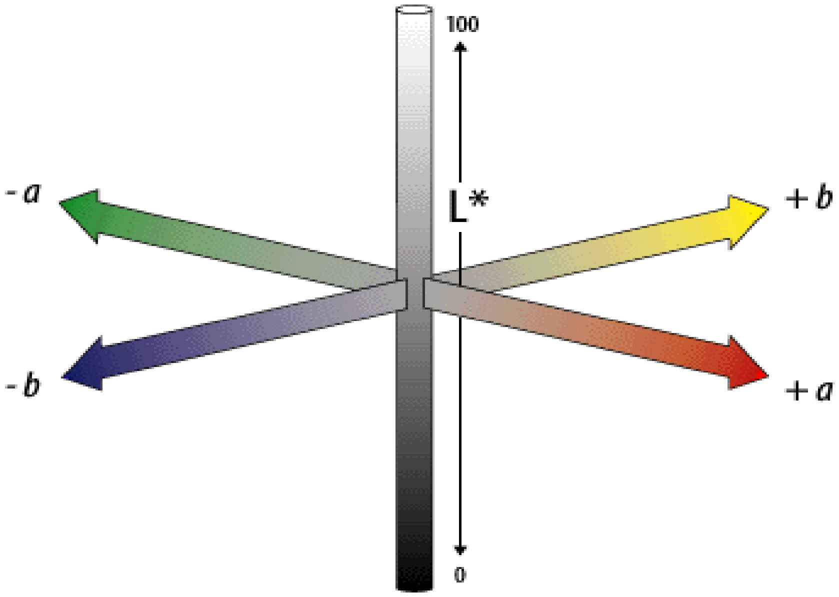






C. (10 Marks) Define color space and its types.

color space



C. Mention applications of ion selective electrode.

Agriculture

- soil samples - determination of calcium, nitrates, sodium, potassium, boron, ammonium and halogenides
- fodder - measurements of nitrogen after Kjeldahlization, calcium, halogenides, sodium, potassium, nitrates, to ensure quality products
- plant tissues - nitrates, halogenides, cyanides, calcium, sodium and potassium can be measured currently
- fertilizers - determination of potassium, calcium, nitrogen and nitrates in fertilizers

Medicine

- blood, serum - measurement of potassium, calcium, sodium, fluorides in samples
- urine - easy determination of fluorides, ammonium and calcium

Food industry

- meat and fish - nitrite and nitrate measurements in meat and fish processing, checking the fluoride level in fish protein to state the toxin concentration
- milk and dairy products - monitoring of fluoride concentrations for checking some toxins
- drinks and juices - determination of the concentration of chlorides, fluorides and carbonates
- alcoholic drinks, beer, wine - determining of potassium, sodium, carbonate, fluoride, and/or bromide levels

Paper industry

- sodium, calcium, silver, sulphide and chloride concentration measurements

Photochemistry

- bromide, silver and nitrate concentration measurements

Geology and mining

- fluoride, chloride and calcium measurements in different types of minerals

Metallurgy and electroplating baths

- concentration measurements of copper, cadmium, free and total cyanides, fluorides, fluoroborates, nitrates and ammonium

Petroleum refining and gas production

- after distillation, the concentrations of ammonia, hydrogen chloride, hydrogen cyanide, hydrogen fluoride and hydrogen sulphide can be determined

- low chloride concentrations in water, used for desalting crude oil, can be determined, too

Medical products

- determination of fluorides in vitamines and tooth pastes, halogenides, copper, nitrates, or, if need be, calcium can be determined in a number of medicaments

Power engineering

- calcium and chloride concentration can be easily measured in boiler and exchanger waters

Water systems

- natural water - calcium, potassium, sodium, silver, lead, cadmium, halogenide, ammonium, sulphide and carbonate ions are currently determined
- drinking water - fluorides and nitrates are most frequently measured substances
- sea water - determination of halogenide, nitrate, potassium and sodium ions
- waste water - concentration measurements of copper, silver, cyanides and ammonium, and, if need be, of nitrogen after Kjeldahlization

Educational system

- at present, the electrodes are an important implement for numerous analytic and instrumental subjects. They help to observe effects and processes concerning the kinetics, equilibrium states and to determine activity coefficients, solubilities and ionizations

Research

- the electrode are used in following fields and branches: food industry, agriculture, agronomy, medicine, chemistry, physics, biology, stomatology, enviroment protection, etc.

Determination of acidity

- In all the mentioned fields and branches, where the ion selective electrodes are used, the glass pH-electrode is also used. The determination of acidity is, in most cases, an essential condition for the qualification of the samples under question.

X-rays of wavelength 1.5405 A are diffracted from crystal at an angle 36^{o}. Assuming that the distance between layers in the crystal (d) =2.44A, (hkl=311) and β= 0.85^{o} calculate the crystal size in nm. (1 nm = 10A).

$$D=0.9\lambda/\beta\cos(\theta)$$

D=11.55 nm

D.) Mention the advantage and disadvantage of ion selective electrode

Advantages and limitations of I.S.E.

Advantages:

1. Linear response: over 4 to 6 orders of magnitude of A.
2. Non-destructive: no consumption of analyte.
3. Non-contaminating.
4. Short response time: in sec. or min. useful in indust. applications.
5. Unaffected by color or turbidity.

Limitations:

1. Precision is rarely better than 1%.
2. Electrodes can be fouled by proteins or other organic solutes.
3. Interference by other ions.

4. Electrodes are fragile and have limited shelf life.

5. Electrodes respond to the activity of uncomplexed ion. So ligands must be absent or masked. m must be kept constant.