Benha university	**4th year students**	**Date : 23.01.2019**
Faculty of science	**Instrumental Analytical chemistry**	**Time: 2 hours**
Chemistry department	**(double course)**	**Code: Ch443**

Answer only four questions:

Q.1: (14 Marks) Derive Lambert-Beer's Law, showing the Instrumental causes deviation from Beers law?

Q.2: (4x3=12 Marks) Discuss each of the following:

1) filter

2) diffraction grating

3) phototubes

4) Determination of pKa of indicator using two methods

Q.3: (12 Marks)

A. (6 Marks) Discuss each of the following:

1) standard addition method

2) Mole-ratio method

B. (6 Marks) 0.35 gram of sample contains cuppous oxalate pentahydrate $[Cu_2(H_2O)_5]C_2O_4$ and oxalic acid. Then, it placed in thermobalance and temperature is raised gradually up to 1000^oC and the final weight of the calcined sample is 0.16 gram due to the formation of cupper oxide. Calculate the purity of cuppous oxalate pentahydrate and cupper in sample. (Cu=63.5, H=1, O=16, C= 12)

Q.4: (12 Marks)

A. (4 Marks) Sketch the diagram of thermogravimetric instrumental (TG and DTA).

B. (4 Marks) Mention the different applications of TG, DTA and DSC tools

C. (4 Marks) Material can behave various behaviors under influence of heat in TG. Show your answer using thermogram.

Q.5: (12 Marks)

A. (4 Marks) Explain the generation of x-ray technique and draw the schematic diagram of x-ray source.

B. (4 Marks) X-rays of wavelength 0.154 nm are diffracted from cubic crystal at an angle 30^{o}. Assuming that n=1, (hkl=221) and $\beta = 0.55^{o}$, calculate the distance (in A) between layers in the crystal, the crystal size (in nm) and crystal lattice (in pm). (1 nm= 10 A = 1000 pm).

C. (4 Marks) Mention different factors affected TG curve (thermogram) and explain only two from them.

Best Wishes,
Dr. Naglaa Mohamed and Dr. Ayman Abdel Razik

C. (6 Marks) 0.35 gram of sample contains cuppous oxalate pentahydrate $[Cu_2(H_2O)_5]C_2O_4$ and oxalic acid. Then, it placed in thermobalance and temperature is raised gradually up to 1000^oC and the final weight of the calcined sample is 0.16 gram due to the formation of cupper oxide. Calculate the purity of cuppous oxalate pentahydrate and cupper in sample. (Cu=63.5, H=1, O=16, C= 12)

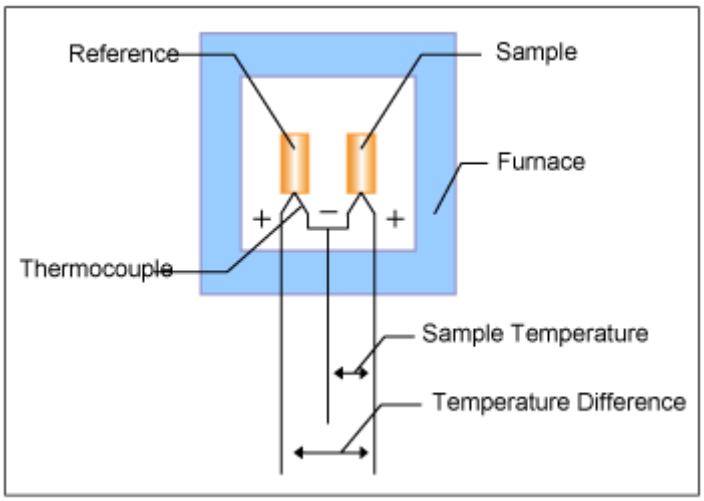
$[Cu_2(H_2O)_5]C_2O_4$ ⟶ CuO Or Cu

Mwt Mwt

X ⟵ 0.16

Purity = (X/0.35) * 100 = %

Sketch the diagram of thermogravimetric instrumental (TG and DTA).



(4 Marks) Mention the different applications of TG, DTA and DSC tools

Determination of transition temperatures (Tg, Tm, Tc, etc.), and the energies involved in the transitions.
Heat of fusion, extents of crystallization, etc.
Glass transition and melting temperatures.
-Kinetics studies (isothermal experiments)
Crystallinity and crystallization rates
Reaction kinetics
-Determination of purity in compounds – by measuring Tm

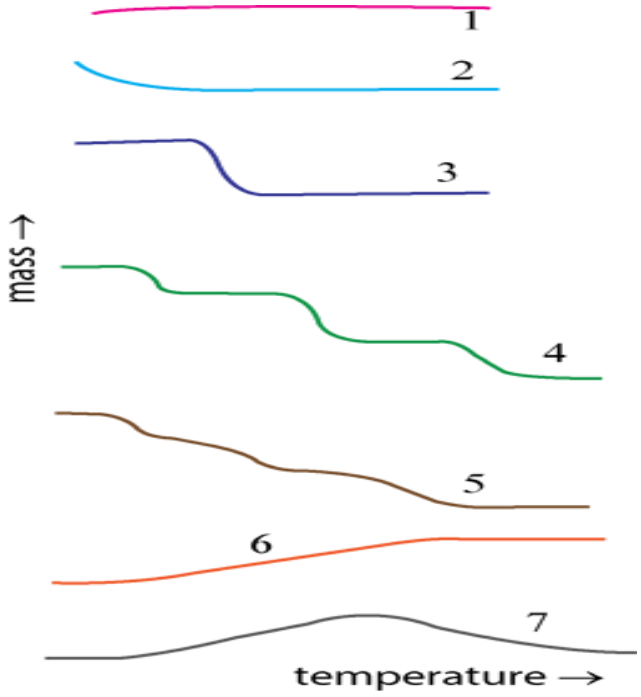
DTA is widely used in the pharmaceutical and food industries
-DTA may be used in cement chemistry, mineralogical research and in environmental studies.
-DTA curves may also be used to date bone remains or to study archaeological materials.

DTA is also widely used in the ceramics and metals industry. The technique is capable of studying high temperature processes (up to 2400oC for some units) and relatively large sample sizes (hundreds of milligrams).

TGA can be used to evaluate the thermal stability of a material. In a desired temperature range, if a species is thermally stable, there will be no observed mass change. Negligible mass loss corresponds to little or no slope in the TGA trace. TGA also gives the upper use temperature of a material. Beyond this temperature the material will begin to degrade. Thermal stability High performance fibers can be compared using TGA as an evaluation of thermal stability.

From the TGA, polyoxazole (PBO) has the highest thermal stability of the four fibers as it is stable up to ca. 500 C.

(4 Marks) Material can behave various behaviors under influence of heat in TG. Show your answer using thermogram.



Explain the generation of x-ray technique and draw the schematic diagram of x-ray source.

X-Rays

Three types of rays emits continuously from a radium material. These rays are known as alpha rays (a rays), Beta rays (p rays) and

gamma rays (x rays). Gamma rays also know as x-rays. The frequency of x-rays as approximately 1020 Hz and its wave length is

approximately 10"10 meter. X-rays are electromagnetic wave which are widely used in medical field and industries for inspection of

human body or any other thing.

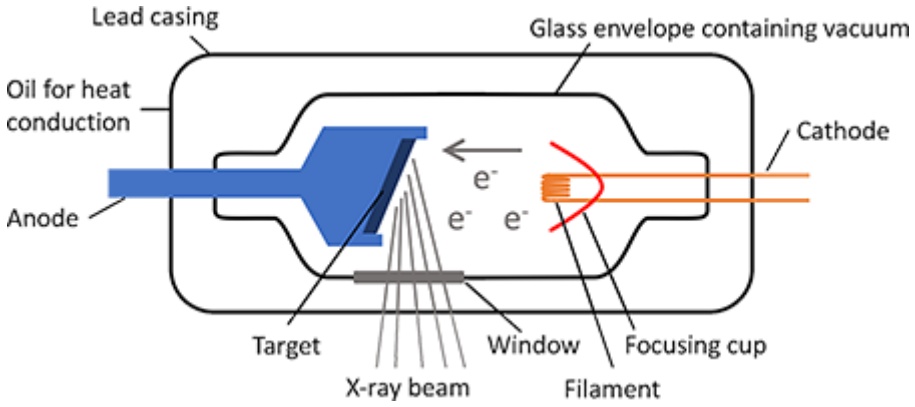
Production of X-rays

X-rays can be produced with the help of high vacuum tube with a heater, cathode and anode. Vacuum tube is operate at very high

voltage. A special electron tube (vacuum tube) is shown in Fig No 11 which is used for production of x-rays. Such a tube has a hot

filament cathode an anode made a very heave metal. Electron flow from the cathode to anode as in any diode tube. However a large

DC voltage is used between cathode and anode of x-rays tube. .



When heater is on and very high anode to cathode voltage is applied the electron emits from cathode and travel toward the anode with very high Velocity, as clear from figure 1, this beam of electron strike the metal anode such speed that new rays are made from the slanting surface of the anode these x-rays seem to bounce sideways ad out thought the well of the tube. As the DC voltage (anode-tocathode of the x-rays tube) is increased, the wavelength of x-rays decreases. Same tubes now operate at more than a million volts.

D. (4 Marks) X-rays of wavelength 0.154 nm are diffracted from cubic crystal at an angle 30^o. Assuming that n=1, (hkl=221) and $\beta = 0.55^o$, calculate the distance (in A) between layers in the crystal, the crystal size (in nm) and crystal lattice (in pm). (1 nm= 10 A = 1000 pm).

1. $n\lambda = d \sin\theta$
2. $a = d/(h^2+k^2+l^2)^{1/2}$
3. $D = 0.9\lambda/\beta\cos\theta$

Mention different factors affected TG curve (thermogram) and explain only two from them.

Variables affecting sensitivity of TA techniques

1. The sample

The chemical description of the sample together with its source, purity and pre-treatments, the particle size may alter the shape of the TA curve, especially where a surface reaction is involved.

2. The crucible

The material of the sample holder or crucible should be stated. Sample holders should not interact with the sample during the course of the experiment.

3. The heating rate (dT/dt)

Experiments may be carried out at slow to very high rates. The 'normal' rate is about 10 K min–1. In order to approach equilibrium conditions most closely, we should use a very small heating rate.

4. The atmosphere

The atmosphere surrounding the sample and its products may greatly affect the measurements. There may be a reaction between the sample and the atmosphere.

5. The mass of the sample

The physical properties, amount and the packing of the sample can affect the results obtained. Small samples are preferable and the average is about 10 mg. Powdered samples or thin films may react more readily than large crystals or lumps.