UNIVERSITI SAINS MALAYSIA

KSCP Examination
Academic Session of 2007/2008

June 2008

EBB 511/3 – Materials Characterisation Techniques

Duration: 3 hours

Please ensure that this examination paper contains <u>FIVE</u> printed pages before you proceed with the examination.

This paper contains SIX questions.

Instruction: Answer **FIVE** questions. If a candidate answers more than five questions, only the first five questions answered will be examined and awarded marks.

Answers to any question must start on a new page.

All questions must be answered in English.

1. [a]   Schematically illustrate the method to measure the inflection temperature in a TG plot.

(20 marks)

[b]   Mathematically describe the procedure to calculate the rate of mass change involved in a TG experiment.

(30 marks)

[c]   Explain in brief the basic requirements that must be fulfilled while designing the furnace for a TG-DTA set up.

(30 marks)

[d]   What are the effects of heating rate and sample mass on the results obtained from a TG-DTA experiment?

(20 marks)

2. [a]   Consider a chemical reaction involving nucleation and growth and obeying the Johnson – Mehl equation, $[-\ln(1-\alpha)]^{1/n} = kt$. Outline the methodology to calculate the activation energy of the reaction through differential and integral approaches.

(50 marks)

[b]   Discuss the importance of temperature measurement and calibration in a thermogravimetric experiment and how the calibration can be done.

(25 marks)

[c]   Discuss in brief how the atmosphere prevailing during a thermogravimetric experiment may affect the TG/DTA results.

(25 marks)

3. [a] Explain the following :

(i) Why do some compounds fluoresce while others do not.

(ii) Why atomic emission spectra consist of discrete line rather than broad bands.

(iii) Why do optical microscope and electron microscope have different resolutions.

(iv) Why do micrographs of SEM appear as 3-dimensional features but only as 2-dimensional with an optical microscope.

(60 marks)

[b] A coloured solution is placed within a UV-visible spectrophotometer. At 465 nm the sample shows an absorbance of 0.79. Calculate the percentage of light that is being absorbed.

(40 marks)

4. [a] A man is facing trial for a hit-and-run case. The prosecutor is trying to convince the jury that the man hit the victim with his car based on an analysis comparing paint chips on the victim's body with the paint on his car (paint on his front bumper). As the expert analyst, you have to explain the following:

(i) Analysis to prove that the paint matches.

(ii) The equipment that you used to do the analysis.

(50 marks)

[b] Infrared spectroscopy such as FTIR and Raman spectroscopy are closely related effects of great utility in identifying molecular species and measuring their concentrations. Discuss the following:

(i) What is the basis of these effects?

(ii) Differences and similarity of the two techniques.

(50 marks)

5. [a] Explain the working principle and applications of an X-ray Fluorescence (XRF).

(40 marks)

[b] A drinking water supply is suspected of being contaminated with lead. Samples of water aspirated directly into an air/acetylene flame gave an absorbance of 0.68 at 283.3 nm. Standard solutions containing 0.5 and 1.0 ppm were found to exhibit absorbances of 0.43 and 0.86, respectively. Assuming that the Beer-Lambert law is obeyed, calculate the concentration of lead within the water sample.

(40 marks)

[c] Why do samples for EM and STM need to be electrically conductive whereas samples for optical microscope and AFM can be conductive or non-conductive?

(20 marks)

6. Discuss for each case the following: how the analysis is done, equipment used and how result is interpreted to solve the issue or problem.

[a] Determination of trace metals present in lubricating oil of turbines that generate electricity.

(25 marks)

[b] A researcher is involved in the study of nano-particles with a core-shell structure. The analysis should include the determination of the particles size and how to confirm the existence of core-shell structure.

(25 marks)

[c] Analysis of a number of components in a sample mixture, i.e calculate concentrations in a Vitamin B group mixture consisting of Vitamin B1 (Thiamine HCl), Vitamin B2 (Riboflavine) and Vitamin B6 (Pyridoxine HCl).

(25 marks)

[d] Characterize effect of sol-gel concentration, pH value, calcination time and temperature on grain size of nano-TiO_2 powders.

(25 marks)

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