CERTIFICATE OF PROFICIENCY IN ASSAYING - THEORY EXAMINATION
WET ASSAYING, INSTRUMENTATION, QUALITY CONTROL AND SAFETY

Total Marks: **180** June 8, 2007
Time Allowed **3 Hours** **All Calculations to be shown**

(Marks)

Q1 **(30)** Answer any **three** of the following:

(a). Describe how a quadrupole mass analyzer works; particularly the role of ac and dc potentials applied to the rods, no need to be quantitative, but you should describe the basic principles underlying the operation of the mass analyzer as a *tunable mass filter*.

(b). A 1.4297g sample of copper concentrate was dissolved using nitric acid and the resulting solution was bulked to 100mL. A 15mL aliquot of this solution was analyzed using the iodide method. If 29.18mL of a 0.04927 M I^- solution was added to reach the end point. Calculate the % copper content of the concentrate.

(c). Explain how the electrode functions in a fluoride ion-selective determination of fluoride. Your answer should include a brief description of membrane function and the mechanism for ion transport across the membrane.

(d). A 0.968 g sample of impure NaOH was dissolved in 200mL of aqueous solution. A 20.0mL portion of this solution was titrated to the equivalence point with 15.8mL of 0.107 M HCl. What is the percentage purity of the original sample?

Q2 **(15)** Describe a step by step procedure for the determination of copper in a chalcopyrite concentrate by electro-gravimetric method. A thorough discussion of possible sources error and how to avoid them is required.

Q3 **(6)** Name the following analytical techniques:

(a). Measurement of the amount of current passing between two electrodes in a chemical cell.

(b). Measurement of current, when voltage is applied to the electrochemical cell.

(c). Measurement of potential difference between two electrodes in an electrochemical cell without any current flow.

Q4 **(15)** Describe, in detail, how to detect and confirm the presence of As, Sn, and Sr in a 5% HNO_3(v/v) solution. Note: Other metals may be present. Only wet classical methods are acceptable.

Q5 **(12)** List the advantages of ICP-MS, compared with ICP-OES, for the analysis of metallic and non-metallic species, and explain the instrumental features that are responsible for these advantages.

Q6 **(10)** In sample preparation process for XRF analysis the accuracy and precision of the results depend on characteristics of the sample that affect fluorescence intensity. Explain how these effects are minimized.

Q7 **(35)** Answer any **five** questions pertaining to atomic absorption and emission.

(a). Strontium determination using nitrous oxide – acetylene flame and atomic resonance line at 460.7 nm gives poor sensitivity. Suggest at least three things that may be tried to improve sensitivity.

(b). A monochromator with a 0.640-m focal length is equipped with a 2400-gr/mm grating. The grating measures 110 mm x 110 mm. Calculate the first-order reciprocal linear dispersion (Å/mm) for this monochromator.

(c). In AAS a major problem in measurement of analyte concentration is the matrix effect where matrix of the sample interferes with the measurement. List and give a short description of four different techniques used to overcome the problem.

(d). For routine work with plasma emission the final solution for analysis is generally prepared in dilute nitric and/or hydrochloric acid. Explain why this final solution is not preferred in phosphoric and/or sulphuric acid?

(e). Define the following: a) signal-to-noise ratio, b) flicker noise, c) shot noise, d) white noise. In each case discuss ways to reduce the noise.

(f). Define the following terms relating to atomic spectroscopy: (1) pressure broadening (2) Doppler broadening (3) sputtering.

Q8 **(15)** Describe briefly how to obtain, any **three**, of the following in solution prior to the determination by ICP-OES of the element mentioned:

(a). Molybdenum oxide in presence of MoS for Molybdenum oxide content.

(b). Mixture of ZnS/ZnO for ZnO contents only (leave ZnS undissolved)

(c). Ilmenite for Ti.

(d). Mine waste-rock for sulphide associated S.

Q9 **(12)** Complete and balance **three** of the following:

1. $K_2S_2O_8 + KBr + H_2SO_4 \rightarrow$ (in buffered weakly alkaline solution)
2. $CuSO_4 + Na_2S_2O_3 + H_2O \rightarrow$
3. $Na_2CrO_4 + KI + HCl \rightarrow$
4. $KMnO_4 + SbCl_3 + HCl \rightarrow$

Q10 **(15)** Answer the following questions regarding Quality Assurance/Quality Control.

(a). Distinguish between the concepts of quality assurance and quality control.

(b). What are Shewhart charts and how do they contribute to QA/QC program?

(c). What criteria are used for the selection of control materials?

Q11 **(15)** Answer any **three** of the following with respect to the WHMIS program:

1. Define the program.
2. Name the participants of the program and their respective responsibilities.
3. What is a WHMIS label?
4. How many types of labels are in use?