

## **1.0 TITLE**

Preparation of Standard Curve and Determination of Heavy Metals in Water and Wastewater Using Atomic Absorption Spectrophotometer

## **2.0 OBJECTIVES**

1. To prepare standard solution of heavy metal
2. To determine standard curve and the concentration of heavy metals in water and wastewater

## **3.0 INTRODUCTION**

Atomic absorption spectrophotometer (AAS) is an analytical technique that use to measures the concentration of elements. Atomic absorption is very sensitive that it can measure down to parts per billion of a gram in a sample. The technique makes use of the wavelengths of light specifically absorbed by an element. It corresponds to the energies needed to promote electrons from one energy level to another, higher, energy level.

Atomic absorption spectrophotometer has variety uses in different area of chemistry such as in clinical analysis, environmental analysis, pharmaceuticals, industry and mining. In environmental analysis, AAS is used to monitor environment conditions by finding out the levels of various elements in rivers, seawater, drinking water, air, petrol, and drinks.

In this experiment, a Polarized Zeeman Atomic Absorption Spectrophotometer (Model Z-5000 Series)(Figure 1) will be operating for water/wastewater analysis. The main unit consists of a lamp chamber, burner, graphite atomizer furnace, monochromator, detector, mechanisms and electrical circuits. In addition, it is provided with a gas controller used to control various gases in flame analysis, power supply for graphite furnace automization and auto sampler.



Figure 1 Polarized Zeeman Atomic Absorption Spectrophotometer (Model Z-5000 Series)

#### 4.0 MATERIALS & METHODOLOGY

60% – 69% Nitric Acid ( $\text{HNO}_3$ )

1000 mg/L copper standard solution

1000 mg/L manganese standard solution

1000 mg/L iron standard solution

1000 mL beaker

250 mL beaker

100 mL volumetric flasks

50 mL volumetric flasks

100 $\mu\text{L}$  micropipette

1000 $\mu\text{L}$  micropipette

500mL measuring cylinder

Vacuum filtration apparatus

De-ionized water

1. Sample preparation:
  - a. Assemble a filtration apparatus.
  - b. Filter 50 mL sample using vacuum pump through a 0.45µm nylon membrane filter/glass fiber filter.
  - c. Collect the filtrate for sample analysis.
2. Standard solution preparation:
  - a. Dilute the standard solution in 100 mL volumetric flask to prepare 10 mg/L stock solution for each element to be measured. Use de-ionized water for dilution.
  - b. Dilute the stock solution in 50 mL volumetric flask to prepare standard working curve for each element.
    - i. Prepare 0 mg/L, 1 mg/L, 2 mg/L and 4 mg/L of copper and iron respectively for working curve preparation.
    - ii. Prepare 0 mg/L, 0.5 mg/L, 1 mg/L and 2 mg/L of manganese for working curve preparation.
  - c. Add a small volume of nitric acid to make the solution acidic (to preserve the solution).
3. The flame atomic absorption spectrophotometer will be set up.
  - a. Place hollow cathode lamps of manganese, iron and copper at right position.
  - b. Make sure supply gas are on for Acetylene (Air-C<sub>2</sub>H<sub>2</sub>) and Compressed Air.
  - c. Switch on power supply, canopy hood, chiller, power supply of spectrophotometer and PC.
  - d. Start of software:
    - i. Double-click the shortcut icon “AA Spectrophotometer” on the screen.
    - ii. Confirm that the atomic absorption spectrophotometer application has started.
    - iii. Click Online status.
    - iv. Click Edit Element
    - v. Save Method

- vi. Click Verify (and switch on Flame on the Spectrophotometer unit)
- vii. Click Auto Zero (wait until green color appear on menu bar)
- viii. Click Ready (wait until green color appear on menu bar)
- ix. Place sample on the sample tray
- x. Click Start for analyze

## 5.0 RESULTS & DISCUSSIONS

Sample (different sources)	Cu, mg/L	Mn, mg/L	Fe, mg/L

Your discussion may include the following questions:

1. What are the preparations should be taken before starting up the equipment? List all the steps and procedures before the equipment can be ran.
2. Why a small volume of nitric acid should be added into the standard solution?
3. Why a sample of water/wastewater should be filtrated prior to analysis?
4. Interpret your results and compare with Standard A or Standard B in Environmental Quality Act 1974.

## 6.0 REFERENCES

Operation Manual: Flame. 2001. Model Z-5000 Series Polarized Zeeman Atomic Absorption Spectrophotometer. 9<sup>th</sup> Edition. Hitachi.

Francis, R. & Annick, R. 2007. Chemical Analysis: Modern Instrumentation Methods and Techniques. 2<sup>nd</sup> Edition. John Wiley & Sons. England.

