DETERMINATION OF HEAVY METALS IN EFFLUENT

WAN MOHD TARMIZI BIN WAN MOHD YUSUF

FACULTY OF SCIENCE

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DETERMINATION OF HEAVY METALS IN EFFLUENT

WAN MOHD TARMIZI BIN WAN MOHD YUSUF

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Name of candidate: **WAN MOHD TARMIZI B WAN MOHD YUSUF** (I.C/Passport No: **861213-29-6015**)

Registration/Matric No: **SGC 110011**

Name of Degree: **MASTER OF SCIENCE (ANALYTICAL CHEMISTRY AND INSTRUMENTAL ANALYSIS)**

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ABSTRACT

The objective of this research project is to determine the concentration of 5 toxic metals from industrial effluent.The five metal elements are cadmium (Cd), lead (Pb), copper (Cu), nickel (Ni), and iron(Fe).Two sources of effluent have been taken for the analysis of heavy metals concentration. It is very important to monitor the concentration of toxic metals effluent whether the concentrations are safe to be released to the environment or need to be treated. A serious contamination to human life and environment can occur if there is no observation on the effluent that will be released to the environment. The sample of effluent used in this work are different from each others depending on their source. The sample preparation need to carefully done to avoid external contamination and loss of target elements. The analysis of heavy metals were performed using AAS and later confirmed by ICP-MS. The results indicate that the effluent have been treated to fit the requirement from Malaysia Enviromental Act.

ABSTRAK

Objektif projek penyelidikan ini adalah untuk menentukan kepekatan 5 logam toksik daripada efluen perindustrian. Lima elemen logam tersebut adalah kadmium (Cd), plumbum (Pb), tembaga (Cu), nikel (Ni), dan besi (Fe). Dua sumber efluen yang telah diambil untuk analisis kepekatan logam berat. Proses ini penting untuk memantau kepekatan logam toksik buangan sama ada kepekatan tersebut adalah selamat untuk dilepaskan ke persekitaran atau perlu dirawat. Pencemaran serius kepada kehidupan manusia dan alam sekitar boleh berlaku jika tiada pemerhatian pada efluen yang akan dilepaskan ke persekitaran itu. Sampel efluen yang digunakan dalam kerja-kerja ini adalah berbeza antara satu sama lain bergantung kepada sumber mereka. Penyediaan sampel perlu dilakukan dengan berhati-hati untuk mengelakkan pencemaran dari luar dan kehilangan unsur-unsur elemen. Analisis logam berat yang dilakukan menggunakan AAS dan kemudiannya disahkan oleh ICP-MS. Keputusan menunjukkan bahawa efluen yang telah dirawat memenuhi keperluan dari Akta Alam Sekitar Malaysia.

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LIST OF SYMBOLS AND ABBREVIATIONS

BOD	Biological Oxygen Demand
COD	Chemical Oxygen Demand
USEPA	U.S Environmental Protection Agency
WHO	World Health Organization
Cd	Cadmium
Pb	Lead
Cu	Copper
Fe	Iron
Ni	Nickel
As	Arsenic
Ca	Calcium
Co	Cobalt
Cr	Chromium
Si	Silicon
Al	Aluminium
Ti	Titanium
V	Vanadium
Zr	Zirconium
Mn	Manganese
Zn	Zinc
Sc	Scandium

FAAS	Flame Atomic Absorption Spectroscopy
AAS	Atomic absorption spectroscopy
HCL	Hollow cathode lamps
PET	Polyethelene
L	Liter
mL	Milliliter
μL	Micro liter
ICP-MS	Induced Coupled Plasma – Mass Spectrometry
μm	Micrometer
HNO_3	Nitric Acid
w/w	Weight/Weight
ppm	Part per million

CHAPTER 1

INTRODUCTION

1.1 WATER

Water in its purest form is odorless, colorless and tasteless. It is in our body, the food we eat and beverages we drink everyday. All living organisms need water to keep on survivng. In some places, it is very valuble and incredibly hard to get. There is no other compund or substance that is more important than water.Water is a molecule that has one oxygen atom and two hydrogen atoms which bonded together by shared electrons. It is a V-shaped polar molecule that negatively charged chraged on oxygen atom and positively charged on hydrogen atoms. This polarity makes water has a strong hydrogen bonding and give special properties to its characteristic such as it is denser in liquid state rather than in solid form.

Water is the only compound that occurs naturally as gas, liquid and solid. It covers almost about 70 percent of the earth surface. However only 3 percent of the water is freshwater. Even more, the distribution of fresh water is not evenly distributed throughout the world. Certain countries do not have enough fresh water to support the growing population and industrial needs in terms of preparing the infrastructure for water treatment and transportation. As example, in China the people in the cities are suffering for water shortages while most of China's river and underground water are mostly polluted.

In all industry, water plays an essential part. It is used as steam to generate a power to run heavy machinery, to cool down the reactor, to clean the waste products and many more. Water is an important ingredients in many products such as chemicals, drugs, household products and many more. Water used in processing foods plant must be absolutely clean and safe for human consumption compare to other usage.

1.2 WATER CONTAMINATION

There are a lot of contributors for water contamination and generally there are two main categories which are direct and indirect sources. Direct sources are coming from industrial wastewater such as refineries, water treatment plants, factories and the others. Every country has its own regulation on waste water quality that must be met before releasing the treated water to the public water source such as river, sea or lake. Indirect sources include all the contaminants which enter the water supply from either groundwater or soils. The residues from manufacturing and agriculture practices which improperly disposed contribute for the contamination.

The contamination in the long run will affect the ecosystem and give variety of bad effects and problems. The drinking water will not be suitable for consumption causing more health problems especially in the third world countries.In Malaysia, there is a regulation called Malaysia's Environmental Law, Environmental Quality Act, 1974, the Malaysia Environmental Quality (Sewage and Industrial Effluents) Regulations, 1979, 1999, 2000. The parameters limits are shown in Table 1.1.

Table 1.1: Parameters Limit of Effluent of Standards A and B (Environmental Quality Act, 1974)

Parameter	Unit	Standard A	B
(1)	(2)	(3)	(4)
(i) Temperature	°C	40	40
(ii) pH Value		6.0 - 9.0	5.5 - 9.0
(iii) BOD at 20°C	mg/l	20	50
(iv) COD	mg/l	50	100
(v) Suspended Solids	mg/l	50	100
(vi) Mercury	mg/l	0.005	0.05
(vii) Cadmium	mg/l	0.01	0.02
(viii) Chromium, Hexavalent	mg/l	0.05	0.05
(ix) Arsenic	mg/l	0.05	0.10
(x) Cyanide	mg/l	0.05	0.10
(xi) Lead	mg/l	0.10	0.5
(xii) Chromium, Trivalent	mg/l	0.20	1.0
(xiii) Copper	mg/l	0.20	1.0
(xiv) Manganese	mg/l	0.20	1.0
(xv) Nickel	mg/l	0.20	1.0
(xvi) Tin	mg/l	0.20	1.0
(xvii) Zinc	mg/l	1.0	1.0
(xviii) Boron	mg/l	1.0	4.0
(xix) Iron (Fe)	mg/l	1.0	5.0
(xx) Phenol	mg/l	0.001	1.0
(xxi) Free Chlorine	mg/l	1.0	2.0
(xxii) Sulphide	mg/l	0.50	0.50
(xxiii) Oil and Grease	mg/l	Nodetectable	10

1.3 WASTE WATER TREATMENT

Waste water treatment is the process to remove contaminants from waste water in all aspect including physical, chemical and biological substance. This is important to make sure the used water is environmentally safe and suitable for disposal or recycle. Currently, it is possible to recycle waste water for drinking water as implemented in Singapore using latest technology. The water with NEWater brand is now been promoted to increase the awareness to use recycle water. The quality of NEWater is unquestionable as it always exceeds the requirements from USEPA(U.S Environmental Protection Agency and WHO (World Health Organization) and it is cleaner than any other water sources in Singapore. This technology however comes with expensive price and not suitable for poor country.

1.4 HEAVY METALS

Heavy metals are elements that have a specific gravity which is five times that of water. They are often found to be the reason for harmful damage to humans in cases leading to environmental pollution from multiple different sources such as mercury, arsenic, lead and cadmium. Some heavy metals such as copper, chromium, iron, zinc and manganese, are necessary for the body but in case there is an over exposure to the same metals, it can lead to heavy metal toxicity symptoms.

Heavy metal toxicity often leads to very dangerous health issues in both adults and children. Long-term exposure can result in diseases like Alzheimer's, neurological degenerative processes, Parkinson's disease, muscular dystrophy, and multiple sclerosis. Heavy metal toxicity may affect the body through various sources. These sources include water, air, food or absorption through the skin when in contact with those exposed to toxic farming, chemical and toxic exposure in industries.

Heavy metals are found in everyday existence and are frequently hard to avoid entirely. Most people can excrete toxic heavy metals from the body successfully. However, some people especially those who suffer from chronic conditions cannot excrete them efficiently enough and a build-up occurs. Recent research also reveals that those who cannot excrete heavy metals efficiently appear to be genetically predisposed to this condition.

1.4.1 CADMIUM

Cadmium (Cd) is a lustrous which reflecting light evenly and efficiently without glitter or sparkle, soft, silver-white and very malleable (ability to deform under compressive stress) metal. The metal is soft enough to be cut with a knife, but it decompose easily when expose to air. It is soluble in acids but not in alkalis. It is similar in many respects to zinc but it forms more complex compounds.

Human uptake of cadmium takes place mainly through food. Foodstuff that is rich in cadmium can greatly increase the cadmium concentration in human bodies. An exposure to significantly higher cadmium level occurs when people smoke. Tobacco smoke transports cadmium into the lungs. Blood will transport it through the rest of the body where it can increase effects by potentiating cadmium that is already present from cadmium rich food. *Itai - Itai disease* is the discovered in Toyama Prefecture, Japan, just after the 2nd World War. There was an increasing number of women ofpost-menopausal age suffered multiple bone fractures and they were brought to thehospital in severe pain. The patients also lost up to 20 cm of body height due to compression fractures of the spine.

It was subsequently discovered that the diseaseoccurred in more than 20% of women above 50 years of age in certain villages along the Jinzuriver in Toyama Prefecture. The distribution of the disease agreed well with the occurrence of high concentrations of cadmium in paddy soil and irrigation water in the sea reas. It was later demonstrated that high concentrations of cadmium in rice correlatedwith the occurrence of the disease and that the patients had increased levels of cadmium in their urine. (Gunnar F., 1995)

Other high exposure can occur with people who live near hazardous waste sites or factories that release cadmium into air and people that work in the metal refinery industry. When people breathe cadmium it can severely damage the lungs. This may even cause death. The biological half-life of Cd is in the range of 10–30 years. In the human body, Cd accumulates in the kidneys and liver in the highest concentration, followed by the pancreas and lungs. There is no known efficient biological mechanism for Cd excretion, thus accumulation in the peripheraltissues is inevitable and generally irreversible.(Amanda C., 2006)

1.4.2 LEAD

Lead (Pb) is a bluish-white lustrous metal. It is very soft, highly malleable, ductile, and a relatively poor conductor of electricity. It is very resistant to corrosion but tarnishes upon exposure to air. Lead isotopes are the end products of each of the three series of naturally occurring radioactive element. Native lead is rare in nature. Currently lead is usually found in ore with zinc, silver and copper and it is extracted together with these metals.

Lead occurs naturally in the environment. However, most lead concentrations that are found in the environment are a result of human activities. Lead is one out of four metals that have the most damaging effects on human health. It can enter the human body through uptake of food (65%), water (20%) and air (15%). Foods such as fruit, vegetables, meats, grains, seafood, soft drinks and wine may contain significant amounts of lead. Lead can enter drinking water through corrosion of pipes.

This is more likely to happen when the water is slightly acidic. That is why public water treatment systems are now required to carry out pH-adjustments in water that will serve drinking purposes. Lead gives no essential function in the human body, it can merely do harm after uptake from food, air or water. It can cause several unwanted effects, such as disruption of the biosynthesis of haemoglobin and anaemia, high blood pressure, kidney damage, miscarriage for pregnant women, disruption of nervous systems, brain damage and many more. Lead can enter a foetus through the placenta of the mother. Because of this it can cause serious damage to the nervous system and the brains of unborn children.Lead (Pb) is well known as an environmental pollutant since it can accumulate in various media, so actual leadexposure reflects both historical and present contaminations.

Levels of lead content in various media have been coupled with data for lead intake and absorption in the humanbody, for both children and adults. It is confirmed that thecritical route of exposure is via ingestion, accounting for 99% of total lead intake, while inhalation contributesonly to 1% of total lead intake. The resulting lead levels in the blood after 2 years of exposure to actualcontamination conditions have been estimated as up to 2.2 μg/dl in children and almost 1 μg/dl in adults.Impacts from lead can occur even at such level. (Mazzimo Pissol, 2009)

1.4.3 COPPER

Copper (Cu) is a reddish metal with a face-centered cubic crystalline structure. It reflects red and orange light and absorbs other frequencies in the visible spectrum due to its band structure, so it as a nice reddish color. It is malleable, ductile, and an extremely good conductor of both heat and electricity. Copper has low chemical reactivity. In moist air it slowly forms a greenish surface film called patina; this coating protects the metal from further attack.

Copper is a very common substance that occurs naturally in the environment and spreads through the environment through natural phenomena. Humans widely use copper in industry, thus the copper quantities in the environment have increased. Rivers are depositing sludge on their banks that is contaminated with copper, due to the disposal of copper-containing wastewater. Copper enters the air, mainly through release during the combustion of fossil fuels. Copper in air will remain there for an eminent period of time, before it settles when it starts to rain. It will then end up mainly in soils. As a result soils may also contain large quantities of copper after copper from the air has settled. Copper can be released into the environment by both natural sources and human activities. Examples of natural sources are wind-blown dust, decaying vegetation, forest fires and sea spray. Long-term exposure to copper can cause irritation of the nose, mouth and eyes and it causes headaches, stomachaches, dizziness, vomiting and diarrhea. Intentionally high uptakes of copper may cause liver and kidney damage and even death. Whether copper is carcinogenic has not been determined yet.There are scientific articles that indicate a link between long-term exposure to high concentrations of copper and a decline in intelligence with young adolescents. Whether this should be of concern is a topic for further investigation.

Copper does not break down in the environment and because of that it can accumulate and bio-magnify in plants and animals when it is found in soils. On copper-rich soils only a limited number of plants has a chance of survival. That is why there is not much plant that can grow near copper-disposing factories. Due to the effects upon plants copper is a serious threat to the productions of farmlands.

1.4.4 IRON

Iron (Fe) is a relatively abundant element in the universe. The sun and many types of stars contain iron in quantity. Iron is found native in a class of meteorites called siderites and it is a minor constituent of the other two classes of meteorites. Iron is the fourth most abundant element in the Earth's crust. The most common iron ore is hematite (Fe_2O_3), from which iron metal is obtained by reduction with carbon. Iron is also found in minerals such as taconite and magnetite, which is commonly seen as black sands along beaches and stream banks.

1.4.5 NICKEL

Nickel (Ni) is a silvery-white, hard, malleable, and ductile metal. It is a good conductor of heat and electricity. In its familiar compounds nickel is bivalent, although it assumes other valences. It also forms a number of complex compounds. Most nickel compounds are blue or green. Nickel dissolves slowly in dilute acids but becomes passive when treated with nitric acid. Finely divided nickel adsorbs hydrogen.

Nickel is a compound that occurs in the environment only at very low levels. Nickel is used for a lot of application in daily life. The most common application of nickel is the use as an ingredient of steal and other metal products and can be found in common metal products such as jewelry. Foodstuffs naturally contain small amounts of nickel. Chocolate and fats are known to contain severely high quantities. Nickel uptake will boost when people eat large quantities of vegetables from polluted soils. Humans may be exposed to nickel by breathing air, drinking water, eating food or smoking cigarettes. Skin contact with nickel-contaminated soil or water may also result in nickel exposure. In small quantities nickel is essential, but when the uptake is too high it can be a danger to human health.

Exposure to the high concentration of nickel will increase the chance of lung cancer, nose cancer, larynx cancer and prostate cancer. Nickel is released into the air by factory by productand will stay inside the soil or flows to the water source after reactions with raindrops. The larger part of all nickel compounds that are released to the environment will adsorb to sediment or soil particles and become immobile as a result. In acidic ground however, nickel is bound to become more mobile and it will often rinse out to the groundwater. Nickel is not the element that accumulate in plants or animals. thus nickel will not bio-magnify up the food chain.

1.5 INSTRUMENTATION

1.5.1 FLAME ATOMIC ABSORPTION SPECTROSCOPY

FAAS (Flame Atomic Absorption Spectroscopy) is a fast and easy technique with an extremely high sensitivity (especially for elements like Pb, Cd, Cu and Cr), although problems can arise as a result of chemical interferences and spectral interferences. The sample is atomized in the flame, through which radiation of a chosen wavelength using a hollow cathode lamp is sent. The amount of absorbed radiation is a quantitative measure for the concentration of the element to be analyzed. Flame atomic absorption spectrometry (FAAS) is one of the most widely used analytical techniques for trace element determination, but it gives poor sensitivity due to the low nebulization efficiency and the short residence time offree atoms in the flame. (Peng Wu, 2009)

Atomic absorption spectroscopy (AAS) is a technique for the quantitative determination of a particular element in a sample. Atomic absorption spectroscopy is based on the absorption of electromagnetic radiation by the excitation of neutral atom due to the high temperature of the flame depending on the mixture of gases used. AAS can be used for the determination of almost 70 different elements in homogenized solution. In AAS systems, analytes are atomized via different atomization techniques which employ air/acetylene and nitrous oxide/acetylene flames. The technique typically makes use of a flame to atomize the sample. Hollow cathode lamps (HCL) are the common radiation source used in AAS system. A nebulizer is used to obtain fine droplets. (S.Bakidere, 2011)

A disadvantage of the AAS technique is the non linearity of the calibration curves when absorbance becomes higher than 0.5 to 1. The relative standard deviations are between 0.3 and 1% for absorbance of 0.1 to 0.2. Detection limits for flame AAS varies from each element. Some elements cannot be measured at all.

1.5.2 ANALYTES AND SENSITIVITY

A FAAS instrument has high sensitivity and the capability to analyze many elements in complex samples. These include the following elements:

Table 1.2: Approximate sensitivity varies from each element (Gary D. Christian, 1994)

Element	**Approximate Sensitivity (ppm)**
As	0.5
Ca	0.1
Cd	0.05
Co	0.2
Cr	0.25
Cu	0.1
Fe	0.15
Pb	0.5

For most of these elements, a Beer's law relationship will hold between approximately 0.5 and 15-20 ppm. This means that FAAS will not be able to determine the concentration of an analyte that is below or above this range. For this reason, the dilution of an unknown sample is frequently required. For example, if the sample contains 20% Fe, then the sample must be diluted so that the Fe concentration is near ~5-15 ppm. Following the analysis of this diluted solution, the original (non-diluted) concentration may be calculated. Figure 1 below shows the schematic diagram of F.AAS on how the process during the analysis occurs within the system.

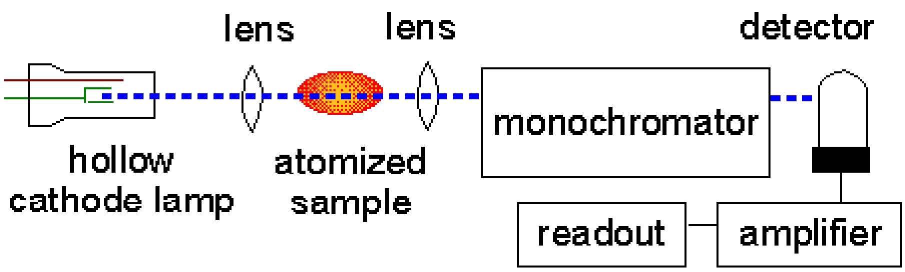

Figure1.1: Schematic Diagram of FAAS (Gary D. Christian, 1994)

1.5.3 TEMPERATURES OF SOME COMMON FLAMES

The gas mixtures used are air/acetylene and nitrous-oxide/acetylene. The latter resulting in higher atomization efficiencies and thus better detection limits for elements like Si, Al, Sc, Ti, V and Zr. The air/acetylene flame can be used for easy atomisable elements such as Arsenic and Selenium. Background correction can be achieved with a deuterium lamp although several disadvantages subsequently occur. Table 1.3 summarize the temperature for each gas mixture used in AAS system.

Table 1.3: Fuel and oxidant mixture in FAAS will give different temperature (Gary D. Christian, 1994)

Fuel	Oxidant	Temperature, K
Hydrogen	Air	2000-2100
Acetylene	Air	2100-2400
Hydrogen	Oxygen	2600-2700
Acetylene	Nitrous Oxide	2600-2800

1.6 SIGNIFICANCE OF STUDY

The content of heavy metals in effluent released to the environment is very important to be controlled and monitored because the effect of heavy metals contamination will be critical in the long run.

1.7 OBJECTIVES OF RESEARCH

The aim of this research is to determine the level of heavy metals intreated effluent released from 2 plating factories in Selangor.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

The effluent from industry must be treated carefully before releasing the water into the water stream. A lot of problems can happen and will effect the ecosystem in a long time before the previous condition can be stablized again by the nature.Some heavy metals are essential for human beings and plants. All of them are toxic if large intakes occur and the main issue is the biomagnifition process for organism within the affected area.

Different elements of heavy metals effect humans in different ways, they can infect the interior organs, weaken bones, carninogen, harm the nerve system and can even cause death. Hence is it necessary to investigate and monitor the content of the hevy metals in effluent released into water stream. Even though the water concentration of heavy metals is low, biomagnifition can occur for living organism living in water stream contaminated with unproperly treated effluent. This will contaminate the water water supply for human daily usage.

2.2 THE HEAVY METALS TOXICITY

Heavy metals are widely distributed and common element in the environment, which does not biodegrade or decay. There are highly toxic element to humans and most otherforms of life. Children, infants and foetuses are at particularly high risk for neurotoxin and developmental effects. The concentrations of heavy metals in the dust, soil, air and water of children’s environments are associated with children’s elevated blood lead levels. As example, lead ingestion by women of child bearing age may impact both the woman’shealth and that ofher foetus, for ingested lead is stored in the bone and released during gestation (Gomaa et al., 2002).

Many industrial plants in developing countries operate without any, or a nominal wastewater treatment and routinely discharge their waste into drains that either contaminate rivers and streams or add to the contaminant load of sewage sludge. Contaminants from industrial, urban, and agricultural sources may enter the food chain in addition to the low water quality of the area. The heavy metals contamination of agricultural soils and crops is causing concerns due to the probable effects on food production and clean water supply for daily usage.

2.3 BIOMAGNIFICATION

There are many different definitions for biomagnifications; Gobaset al.1999 stated that it is the process by which chemical contaminants are concentrated at levels that exceed chemical equilibrium from dietary absorption of the chemical.

Heavy metals are not only toxic for plants. They are also toxic for human beings. They are all toxic if larger amounts are ingested or inhaled. The main issues with heavy metals are the biomagnification due to long residence times. The residence time of cadmium in the soil has a range of 75-380 years. Lead and zinc have residence times of

1000-3000 years. However, half-life for cadmium is 15-1100 years and 740-5900 years for lead. Eventhough the ranges are wide in different soil conditions it is clear that heavy metalpollution is a very long-term matter (Alloway, 1995).

Bioaccumulation of chemical substances can be caused bybiomagnification, mainly via respiratory membranes, or by biomagnifications via dietary uptake. As partitioning between water or food and outer membranes of organisms represents the most important process of biomagnification, it is of particular interest when dealing with substances with certain physicochemical properties, e.g., lipophilicity and persistence. In aquatic systems, sediments, and soils, these substances tend to concentrate mainly in the lipid fraction of organisms and may lead to substantial physiological burdens. The accumulation of such residues in the food chain can reach levels toxic to predators and represents a risk for human health. Assessing the biomagnifications potential is therefore an important issue for the environmental and human risk assessment of chemicals and one of the main features in environmental monitoring.

Biomagnificationof these metals by aquatic organisms is expectedto take place and molluscs are one of the organisms that are efficient in biomagnification of heavymetals in the aquatic ecosystems (Roper et al., 1996). Sg. Sarawak has been the source for the fishermen to catchfish, prawns and molluscs to the residents living along the river corridors. It is suspected that theorganism from Sg. Sarawak Kanan, particularly inareas near Bau and Sg. Bau, will be highly contaminated with heavy metals, hence endangering the health of those who consume them.

In this study, three edible molluscs species were screened for heavy metal content. The objective of this study was to determine the suitability, interms of health and safety, of three molluscspecies, namely *Clithon sp. nr rectropictus*, *Brotiacostula* and *Melanoidestuberculata*, from Sg.Sarawak Kanan as sources of food and to determine the potential of using these three molluscspecies as biomonitors for heavy metal pollutionin freshwater ecosystems. Further biomagnification of metals will be experienced as it moves up the food chain (S.Lau, 1998).

2.4 THE EFFLUENT ANALYSIS

Trace metals are considered to be major toxicant in contaminated water worldwide (Chi-Man and Jiu, 2006; Katsoyiannis and Katsoyiannis, 2006; Asonyeet al., 2007 and Yasuhiro et al., 2007). Several studies have been attempted assessing heavy metal pollution according to the distribution of particle size and to relationship of its organic content (Hiraizumiet al., 1978; Kristensen, 1982 and Simokawaet al., 1984).

Heavy metal levels in many natural water bodies across the world have been investigated. Cooper et al. (1978) analyzed water quality of the river Tean Staff and found an increase in cadmium levels with addition of sewage. Polprasert (1982) determined heavy metal levels in water of the Chao Phraya river estuary, Thailand and discussedtheir long term impact on the aquatic environment.

Mart and Nurnberg (1984) determined trace metal levels in the eastern Arctic ocean while Abaychi and Douabul(1985) determined trace metals in Shatt Al-Arab river, Iraq and indicated metal levelsto be within the recommended limits. Maroofet al. (1986) determined cadmium andzinc concentrations in drinking water supplies of Dhaka city, Bangladesh andhighlighted the impact of addition of bleaching powder and pumping on zincconcentration. Jing and Wei-Wen (1988) determined the concentration of trace metals in the Qiantang-Jiang river and its estuary Southern China and found higher levels of metals with addition of industrial wastes. Pelig-Ba et al. (1991) determined trace metals concentrations in Borehole waters from the upper regions and the Accra plains of Ghana.

Vazquez et al. (1998) determined dissolved metals in Alvarado lagoon, Maxico and examined the seasonal variations in the levels of cadmium, copper and lead. Ozmenet al. (2004) conducted a preliminary study on heavy metal (Zn, Mn, Ni, Cu, Cr, Co and Pb) concentrations in surface water of Hazar lake and discussed the heavy metal pollution status of the lake. Emoyanet al. (2005) evaluated heavy metals loading of river Ijana, Nigeria. Their results indicated higher metal contents in winterseason. Thariet al. (2005), in a multivariate analysis of heavy metal concentration insoil, sediment and water in the region of Meknes (Central Morocco), compared themetal contents in water and sediment to suggest correlations between them.

Abuludeet al. (2006) determined Fe, Cr, Cd, As, Ni, Co and Zn in drinking water samples in Akure, Nigeria. Adefemiet al. (2008) determined heavy metal (Zn, Pb, Mn, Fe, Cu,Co, Cr, Cd and Ni) contents in water from Ureje dam in south-western Nigeria in order to assess the water quality. The impact of heavy metal inputs from various industries has been investigated in several studies.

Huynh-Ngoc et al. (1988) determined cadmium levels in the Rhoneriver polluted by industrial wastes. Peerzadaet al. (1990) studied distribution of heavymetals in Gove harbour, northern territoty, Australia to find the impact of a bauxitetreatment plant on the heavy metal status of water. Vazquez et al. (1993) investigatedheavy metals to study effects of industrial lead inputs into the San Andres lagoon,Tamaulipas, Maxico. They carried out a comparative study of several metals (Cd, Co,Cu, Fe, Mn, Ni, and Zn). Sahet al. (2000) conducted a study on assessment of heavy metal pollution of water in the Narayani River, Nepal contaminated by paper industry effluents. Sanayeiet al. (2009) analyzed heavy metal levels in Zayandeh Rood River, Isfahan-Iran at seven sites to observe the influence of the industrial activities and dump of municipal waste on heavy metal concentrations in this region.

CHAPTER 3

METHODOLOGY

3.1 EFFLUENT SAMPLE

The samples for effluents were taken from 2 different plating factories (Sample A) and (Sample C) both located in Selangor.

3.2 SAMPLING

Sample A and Sample C were collected at the effluent discharge point to water stream that flows directly into the drain and finally the lake or river. The effluents samples were collected from flowing water stream and kept in 1 L clean polyethylene (PET) bottle. The effluent was acidified with diluted nitric acid to preserve its condition before analysis.

3.3 MATERIALS

3.3.1 CHEMICALS

a) 20 % (w/w) nitric acid from Merck.

b) Stock solution 1000 ppm from Merck.(Cadmium and lead).

c) Stock solution 1000 ppm from Spectrosol. (Copper nitrate, iron nitrate and nickel nitrate).

d) Stock solution multi-element calibration standard 10 ppm from Agilent.

3.3.2 APPARATUS

a) 1 L clean polyethylene (PET) bottle.

b) 50 mL and 100 mL volumetric flask.

c) Auto pipette (100-1000 uL).

d) 50 mL and 100 mL beaker.

e) Plastic pippette

3.3.3 INSTRUMENT

a) AAS Atomic Spectroscopy Spectrophotometer (Perkin Elmer AAanalyst 400).

b) ICP-MS Induced Coupled Plasma – Mass Spectrometry (Agilent 7700).

3.4 METHODOLOGY

3.4.1 INSTRUMENTATION

The instrument was set up with manufacturer's guidelines for optimal performance. The hallow cathode lamp is warmed up for 15 minutes prior to analysis. Table 3.1 shows the setting for the instrument.

Table 3.1: AAS setting for analysis

Wavelength	**228.8 nm**
Background Correction	D_2
Signal type	Continuous
Measurement Time	40 Seconds
Measurement Mod	Absorption
Bandpass	0.5 nm
Lamp Current	50%
Flame Fuel	Air-C_2H_2
Fuel Flow	0.8 L/min
Nebulizer Uptake	4 seconds
Burner Height	7.4 cm

Every element has multiple wavelength where it can be analysed using AAS. The best wavelength has been selected for good signal and less interference. Table 3.2 shows the selected wavelength for each element.

Table 3.2: The selected wavelength for the tested elements

Element	**Wavelength (nm)**
Cadmium (Cd)	228.802
Lead (Pb)	220.353
Nickel (Ni)	231.604
Iron (Fe)	338.204
Copper (Cu)	327.393

3.4.2 SAMPLE ANALYSIS

Effluent sample was filtered filtration through 0.45µm membrane filter and was transferred into a 50 mL volumetric flask.Transfer the sample into a 100 mL beaker and add 1 mL concentrated HNO_3. Heat slowly without boiling on the suitable hot plate and adjust the temperature accordingly. After all the visible solids are digested with the water, cool down the sample.Transfer the sample back to 50 mL volumetric flask and top up with distilled water if needed. Analyze the samples using flame AAS. (APHA 3030 B, 2005)

A calibration curve was made using 3 points of different standard for each element by linear regression analysis of absorption intensity versus standard concentration. The standard concentrations were different for each element depending on suggested concentration by Perkin Elmer. Linearity was evaluated by calculating correlation coefficient. A quality control check was used after calibration to check whether the calibration curve can give reliable and accurate result.

The standard solutions were freshly prepared for each analysis to avoid any error as the solutions will be diluted several times from the stock solution. The standard operating procedure figure 3.1 is shown below.

The analysis was carried further using ICP-MS to confirm the test result with lower standard solution to confirm the results analysis.

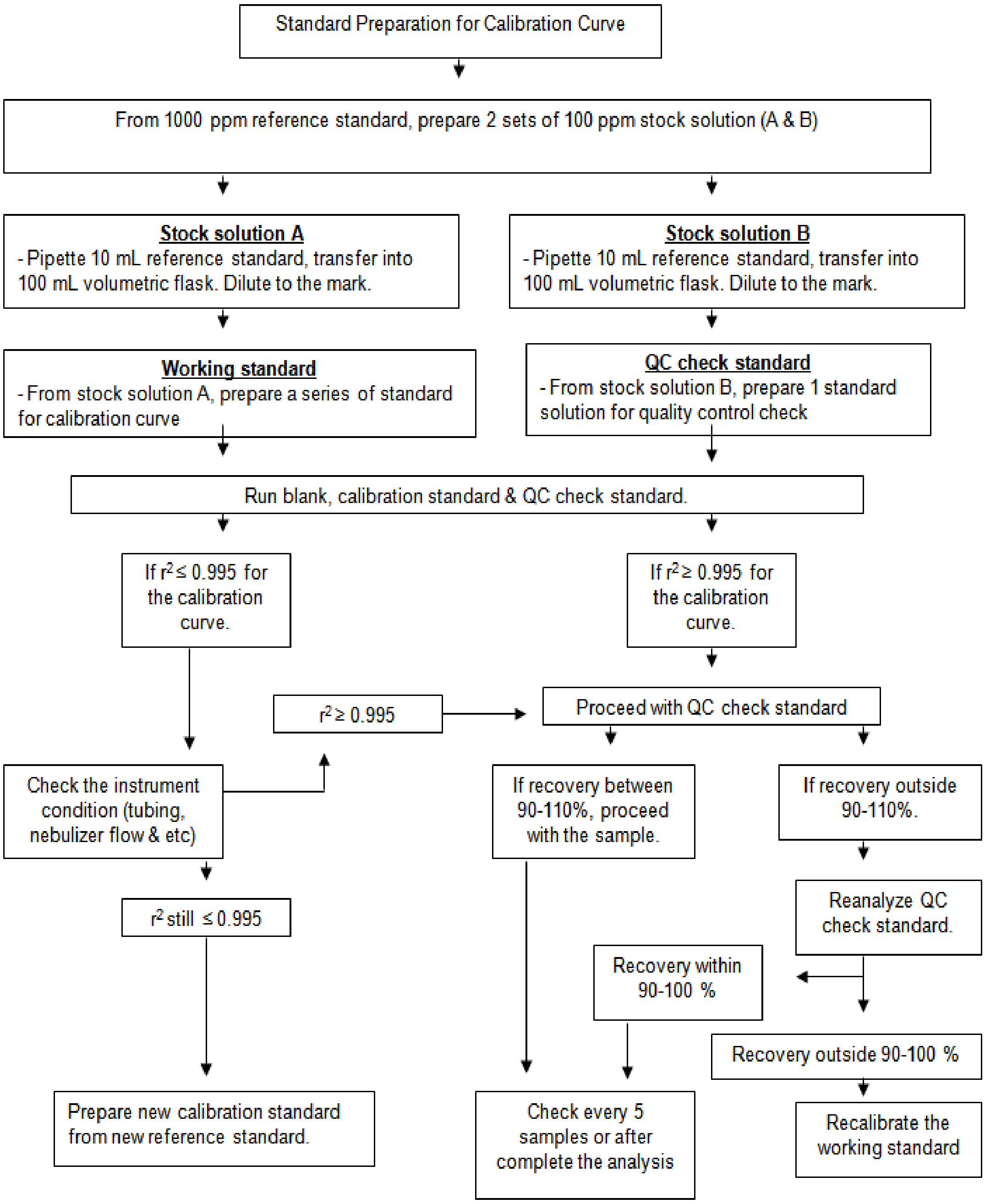

Figure 3.1: Standard operation procedure for AAS/ICP-MS operation

CHAPTER 4

RESULTS AND DISCUSSION

4.1 METHOD VALIDATION

4.1.1 CALIBRATION CURVE

The analysis was done using freshly digested effluent and freshly prepared standard solution. Most instrumental methods of analysis are relative such as in spectrophotometer, it measures the fraction of electromagnetic radiation from a light source that is absorbed by the sample. This electromagnetic radiation need to be related to the analyte concentration which means that the instrument needs to be calibrated every batch of the analysis.

Calibration is made by preparing a series of standard solutions for the analyte of interest at known concentration and measuring the instrument response for each single standard that will be represented as a point to create an analytical calibration curve of response versus concentration. Using the curve, an unknown sample concentration can be determined from the response produced from the sample.

Table 4.1: The calibration curve for Cd, Pb, Ni, Fe and Cu for AAS analysis.

Element	Correlation Coefficient (R^2)	Intercept	Slope
Cadmium (Cd)	0.984	0.127	0.282
Lead (Pb)	0.987	0.015	0.021
Nickel (Ni)	0.997	0.004	0.202
Iron (Fe)	0.999	0.001	0.108
Copper (Cu)	0.997	0.004	0.202

The correlation coefficient, R_2 values for the calibration curves for all the elements are more than 0.98 which are good and reliable curve. It is important to check the best concentration for the instrument to analyze the element. Certain elements like Cd and Cu give unreliable results at the concentration of more than 3 ppm. According to Peng Wu et. al, (2004), this is one of disadvantage of using AAS for trace metal analysis due to low efficiency of nebulisation and short time for free atoms excitation in the flames. While for Pb, low concentration of analyte will not give a sensitive and accurate result.

Quantitative measurements in atomic absorption are based on Beer's Law, which states that concentration is proportional to absorbance (C = kA). It is well known, however, that for most elements,particularly at high concentrations, the relationship between concentration and absorbance deviates from Beer's Law and is not linear. There are several reasons for this, including stray light, humidity of temperature and space in the absorbing cell, line broadening, and in some cases, absorption at nearby lines.

According to W. B. Barnett et. al, (1984), a calibration curve defined using this equation is forced to go through zero absorbance and zero concentration. A least squares technique is used to determine the K1 coefficient when two or more standards (maximum = 8) are used for calibration. K0 is the reslope coefficient, which is set to 1.0 during initial calibration. Figure 4.1 to 4.5 shows the calibration curve for the analysis.

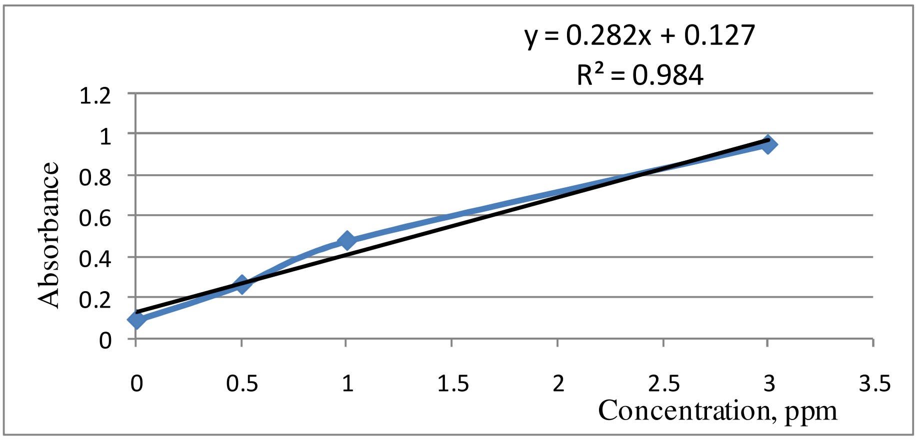

Figure 4.1: Cd calibration curve

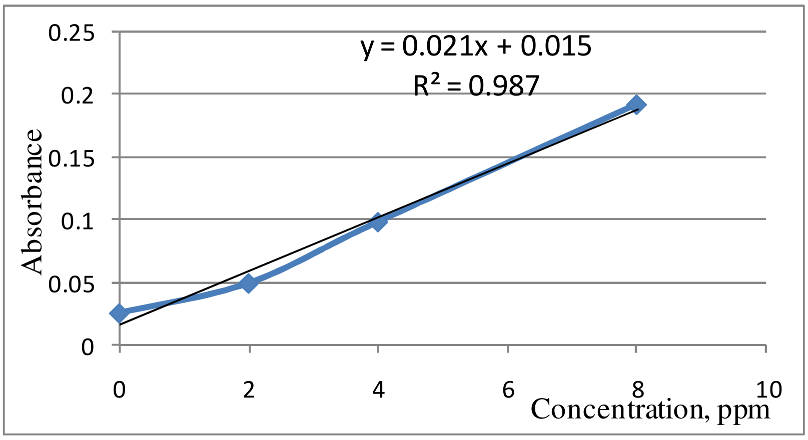

Figure 4.2: Pb calibration curve

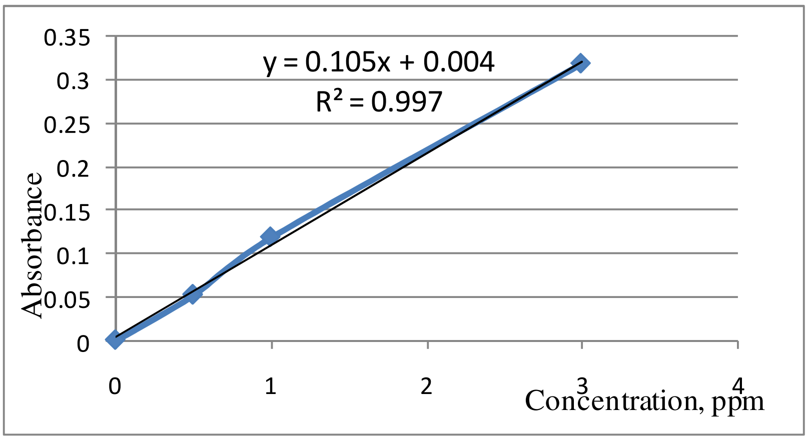

Figure 4.3: Ni calibration curve

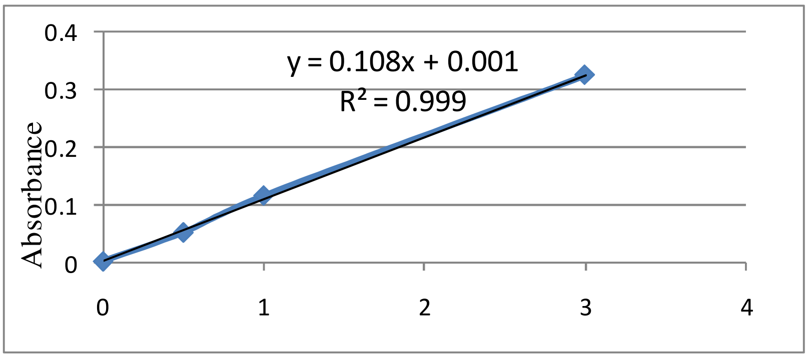

Figure 4.4: Fe calibration curve

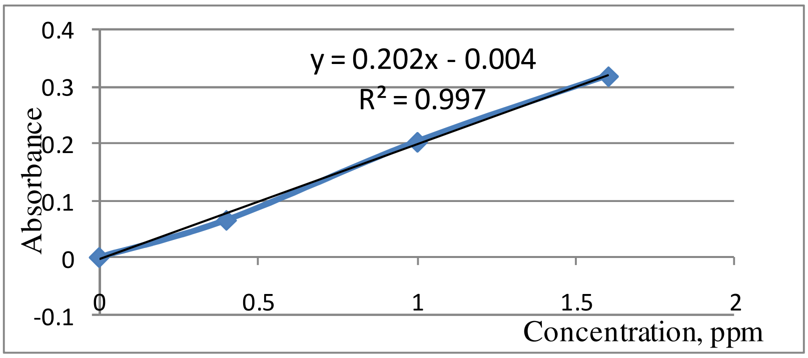

Figure 4.5: Cu calibration curve

The result from Table 4.2 shows that the level of heavy metals released by both company is lower than Malaysia Environmental Quality (Sewage and Industrial Effluents) Regulations. Both factories have succeeded to treat the effluent from their waste before releasing to the environment. Since the analysis was done using AAS, the sensitivity is accurate from around 0.2 ppm to higher concentration (Perkin Elmer Corporation, 1994). The analysis was carried further using ICP-MS with lower standard solution. The results are lower than the lowest standard used in each analysis as shown in Table 4.2.

Table 4.2: The analysis results for effluent samples using AAS.

	Sample A	**Sample C**	**Lowest Standard Solution, ppm**
Cadmium **(Cd)**, ppm	Not Detected	Not Detected	0.5
Lead **(Pb)**, ppm	Not Detected	Not Detected	2
Iron **(Fe)**, ppm	Not Detected	Not Detected	0.5
Nickel **(Ni)**, ppm	Not Detected	0.006	0.5
Copper **(Cu)**, ppm	Not Detected	0.004	0.4

Both factories have succeeded to treat the effluent from their waste before releasing to the environment. The analysis was carried further using ICP-MS with lower standard solution.

The results from ICP-MS with lower standard solution showed that the heavy metals of interest in still undetectable except for Ni from Sample A which is 0.01 ppm. However the maximum limit for Ni according to Malaysia Environmental Quality (Sewage and Industrial Effluents) Regulations is 0.2 ppm.

Table 4.3: The calibration curve for Cd, Pb, Ni, Fe and Cu ICP-MS analysis.

Element	**Correlation Coefficient (R^2)**
Cadmium (Cd)	0.999
Lead (Pb)	0.995
Nickel (Ni)	0.999
Iron (Fe)	0.996
Copper (Cu)	0.999

Table 4.4: The analysis results for effluent samples using ICP-MS.

	Sample A	**Sample C**	**Lowest Standard Solution**
Cadmium (**Cd**), ppm	Not Detected	Not Detected	0.02
Lead (**Pb**), ppm	Not Detected	Not Detected	0.02
Iron (**Fe**), ppm	Not Detected	Not Detected	0.02
Nickel (**Ni**), ppm	0.01	Not Detected	0.02
Copper (**Cu**), ppm	Not Detected	Not Detected	0.02

4.2 INTERFERENCES

Atomic absorption analysis technique has several interferences. The interferences in ASS can be categorized in six categories which are chemical interferences, ionization interferences, matrix interferences, emission interferences, spectral interferences, and background absorption. The common interferences are chemical, ionization and matrix.

In this research, since the sample matrix is a treated effluent with very low content of suspended solid and clear colour we can neglect the possibility of interferences during the analysis.(Gary D. Christian, 1994). ICP-MS analysis does not affected by all these interferences since the plasma within the system can provide enough heat to ionize the sample to remove or minimize the interferences and with the help of the sophisticated hardware and software the ICP-MS result is more accurate and reliable.

4.3 ACCURACY AND REPEATABILITY

Accuracy is the degree of agreement between the measured value and the true value. An absolute true value is seldom known. Using a good analytical technique to make a comparison against a known standard sample, the accuracy can be determined.

While repeatability is important to check the capability of instrument to produce reliable results after different runtime. This can be used to determine any random and systematic error during the standard solution preparation or operation between different operators.

The result replicates for Cd in Table 4.5 show good consistency and accuracy. The quality control sample prepared by different standard solution is used to reduce the error. The recovery of 99.15 % can be used as an indicator whether the result analysis is acceptable and reliable. While for Pb analysis in Table 4.6, the mean recovery is 99.72% which is good and RSD is 0.422. The results are less reproducible but still acceptable. Analysis of Ni as in table 4.7 gave the accuracy of 99.15% and the reproducibility is still acceptable. Fe QC check gave higher results probably due to some trace contamination from glassware or distilled water. The recovery is 104.85 %. Cu accuracy and reproducibility tests have the lowest recovery, 95.50 % and low reproducibility, RSD 1.235. This is probably due to low level of analyte concentration and contamination from glassware or distilled water.

Table 4.5: Replicates of 2.000 mg/L Cd prepared from stock reference standard

	Cadmium 2.000 (mg/L)
Replicate 1	1.979
Replicate 2	1.984
Replicate 3	1.979
Replicate 4	1.980
Replicate 5	1.985
Replicate 6	1.980
Replicate 7	1.988
Replicate 8	1.989
Replicate 9	1.980
Replicate 10	1.982
Mean	4.986
Recovery (%)	99.15
SD	0.004
RSD	0.193

Table 4.6: Replicates of 5.000 mg/L Pb prepared from stock reference standard

	Lead 5.000 (mg/L)
Replicate 1	4.966
Replicate 2	4.995
Replicate 3	4.972
Replicate 4	4.976
Replicate 5	4.998
Replicate 6	4.997
Replicate 7	4.989
Replicate 8	4.988
Replicate 9	4.981
Replicate 10	4.997
Mean	4.986
Recovery(%)	99.72
SD	0.016
RSD	0.422

Table 4.7: Replicates of 2.000 mg/L Ni prepared from stock reference standard

	Nickel 2.000 (mg/L)
Replicate 1	1.979
Replicate 2	1.984
Replicate 3	1.979
Replicate 4	1.980
Replicate 5	1.985
Replicate 6	1.980
Replicate 7	1.988
Replicate 8	1.989
Replicate 9	1.980
Replicate 10	1.982
Mean	1.983
Recovery(%)	99.15
SD	0.004
RSD	0.193

Table 4.8: Replicates of 2.000 mg/L Fe prepared from stock reference standard

	Iron 2.000 (mg/L)
Replicate 1	2.087
Replicate 2	2.087
Replicate 3	2.082
Replicate 4	2.099
Replicate 5	2.098
Replicate 6	2.108
Replicate 7	2.102
Replicate 8	2.099
Replicate 9	2.114
Replicate 10	2.090
Mean	2.097
Recovery(%)	104.85
SD	0.010
RSD	0.485

Table 4.9: Replicates of 0.3 mg/L Cu prepared from stock reference standard

	Copper 0.400 (mg/L)
Replicate 1	0.372
Replicate 2	0.381
Replicate 3	0.381
Replicate 4	0.380
Replicate 5	0.385
Replicate 6	0.384
Replicate 7	0.382
Replicate 8	0.390
Replicate 9	0.380
Replicate 10	0.386
Mean	0.382
Recovery(%)	95.50
SD	0.005
RSD	1.235

The result replicates for Cd in table 4.5 to table 4.9 show good consistency and accuracy. The quality control sample prepared by different standard solution is used to reduce the error.

As for ICP-MS analysis accuracy check, the sample quality control will be analyzed twice which are after the calibration has been constructed and after the analysis of the sample. The concentration of the SQC is 0.12 ppm. ICP-MS gave accurate result and only gave deviation within 0.01 ppm. Table 4.10 summarize the analysis.

Table 4.10: Analysis of SQC sample (0.12 ppm)

	SQC 1	**SQC 2**	**SQC value**	**Deviation (+/-)**
Cadmium **(Cd)**, ppm	0.13	0.13	0.12	0.01
Lead **(Pb)**, ppm	0.12	0.12	0.12	0.00
Iron **(Fe)**, ppm	0.12	0.11	0.12	0.01
Nickel **(Ni)**, ppm	0.12	0.12	0.12	0.00
Copper **(Cu)**, ppm	0.12	0.12	0.12	0.00

CHAPTER 5

CONCLUSION AND RECOMMENDATION

The results show that the concentration of the heavy metals are under the permitted value and can be safely released to the environment. This monitoring should be done regularly as must be done as a guideline for effluent treatment. This study has covered the determination of 5 common toxic heavy metals in industrial effluent. In order to improve the accuracy and sensitivity, the analysis need to be performed using Graphite Furnace AAS or ICP-OES/MS. Even AAS can be used to detect at low level concentration around 1 ppm, there is a need to determine the metal concentration at lower level since the new technology keep on advancing in our daily life, various complex compounds containing toxic metal flows into our water stream as effluent generated by industry. Both factories have managed to treat their waste water before releasing the effluent into the environment.

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Method Loaded
Method Name: Cd in Effluent
Method Description: Cd in Effluent
Method Last Saved: 5/18/2013 10:57:05 AM

Sequence No.: 3
Sample ID: Blank
Analyst:
Autosampler Location:
Date Collected: 5/18/2013 10:57:08 AM
Data Type: Original

Replicate Data: Blank

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[0.00]	0.088	10:57:09	Yes
2		[0.00]	0.089	10:57:13	Yes
3		[0.00]	0.088	10:57:18	Yes
4		[0.00]	0.088	10:57:22	Yes
5		[0.00]	0.088	10:57:26	Yes
6		[0.00]	0.088	10:57:31	Yes
7		[0.00]	0.088	10:57:35	Yes
8		[0.00]	0.088	10:57:39	Yes
9		[0.00]	0.089	10:57:43	Yes
10		[0.00]	0.088	10:57:48	Yes
Mean:		[0.00]	0.088		
SD:		0.00	0.0003		
%RSD:		0.00	0.36		

Auto-zero performed.

Sequence No.: 4
Sample ID: Calib Std 1
Analyst:
Autosampler Location:
Date Collected: 5/18/2013 10:58:10 AM
Data Type: Original

Replicate Data: Calib Std 1

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[0.5]	0.260	10:58:11	Yes
2		[0.5]	0.262	10:58:15	Yes
3		[0.5]	0.261	10:58:20	Yes
4		[0.5]	0.261	10:58:24	Yes
5		[0.5]	0.261	10:58:28	Yes
6		[0.5]	0.260	10:58:32	Yes
7		[0.5]	0.261	10:58:36	Yes
8		[0.5]	0.262	10:58:41	Yes
9		[0.5]	0.261	10:58:45	Yes
10		[0.5]	0.259	10:58:49	Yes
Mean:		[0.5]	0.261		
SD:		0.0	0.0008		
%RSD:		0.0	0.29		

Standard number 1 applied. [0.5]
Correlation Coef.: 1.000000 Slope: 0.52190 Intercept: 0.00000

Sequence No.: 5
Sample ID: Calib Std 2
Analyst:
Autosampler Location:
Date Collected: 5/18/2013 10:59:06 AM
Data Type: Original

Replicate Data: Calib Std 2

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[1]	0.480	10:59:07	Yes
2		[1]	0.477	10:59:11	Yes
3		[1]	0.481	10:59:16	Yes

4	[1]	0.476	10:59:20	Yes
5	[1]	0.479	10:59:24	Yes
6	[1]	0.476	10:59:29	Yes
7	[1]	0.481	10:59:33	Yes
8	[1]	0.477	10:59:37	Yes
9	[1]	0.479	10:59:42	Yes
10	[1]	0.478	10:59:46	Yes
Mean:	[1]	0.478		
SD:	0	0.0018		
%RSD:	0	0.37		

Standard number 2 applied. [1]
Correlation Coef.: 1.000000 Slope: 0.57402 Intercept: 0.00000

==

Sequence No.: 6
Sample ID: Calib Std 3
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:00:14 AM
Data Type: Original

Replicate Data: Calib Std 3

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[3]	0.952	11:00:16	Yes
2		[3]	0.951	11:00:20	Yes
3		[3]	0.952	11:00:24	Yes
4		[3]	0.952	11:00:28	Yes
5		[3]	0.958	11:00:33	Yes
6		[3]	0.952	11:00:37	Yes
7		[3]	0.954	11:00:42	Yes
8		[3]	0.955	11:00:46	Yes
9		[3]	0.951	11:00:50	Yes
10		[3]	0.957	11:00:55	Yes
Mean:		[3]	0.953		
SD:		0	0.0024		
%RSD:		0	0.25		

Standard number 3 applied. [3]
Correlation Coef.: 1.000000 Slope: 0.56123 Intercept: 0.00000

Calibration data for Cd 228.80 **Equation: Nonlinear Through Zero**

ID	Mean Signal (Abs)	Entered Conc. mg/L	Calculated Conc. mg/L	Standard Deviation	%RSD
Blank	0.0000	0	0.000	0.00	0.4
Calib Std 1	0.2609	0.5	0.500	0.00	0.3
Calib Std 2	0.4785	1.0	1.000	0.00	0.4
Calib Std 3	0.9533	3.0	3.000	0.00	0.3

Correlation Coef.: 1.000000 Slope: 0.56123 Intercept: 0.00000

==

Sequence No.: 7
Sample ID: SQC 2 ppm
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:01:30 AM
Data Type: Original

Replicate Data: SQC 2 ppm

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	1.979	1.979	0.777	11:01:31	Yes
2	1.984	1.984	0.778	11:01:36	Yes
3	1.979	1.979	0.777	11:01:40	Yes
4	1.980	1.980	0.777	11:01:44	Yes
5	1.985	1.985	0.778	11:01:49	Yes
6	1.980	1.980	0.777	11:01:53	Yes
7	1.988	1.988	0.779	11:01:58	Yes
8	1.989	1.989	0.779	11:02:02	Yes
9	1.980	1.980	0.777	11:02:06	Yes

10	1.982	1.982	0.778	11:02:11	Yes
Mean:	1.983	1.983	0.778		
SD:	0.004	0.004	0.0009		
%RSD:	0.193	0.193	0.11		

==

Sequence No.: 8
Sample ID: Sample A
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:02:38 AM
Data Type: Original

Replicate Data: Sample A

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	-0.014	-0.014	-0.008	11:02:39	Yes
2	-0.007	-0.007	-0.004	11:02:44	Yes
3	-0.013	-0.013	-0.007	11:02:48	Yes
4	-0.010	-0.010	-0.006	11:02:52	Yes
5	-0.015	-0.015	-0.008	11:02:57	Yes
6	-0.003	-0.003	-0.002	11:03:01	Yes
7	-0.016	-0.016	-0.009	11:03:05	Yes
8	-0.007	-0.007	-0.004	11:03:10	Yes
9	-0.016	-0.016	-0.009	11:03:14	Yes
10	-0.010	-0.010	-0.006	11:03:18	Yes
Mean:	-0.011	-0.011	-0.006		
SD:	0.004	0.004	0.0024		
%RSD:	39.09	39.09	39.13		

==

Sequence No.: 9
Sample ID: Sample C
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:03:34 AM
Data Type: Original

Replicate Data: Sample C

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	-0.001	-0.001	-0.001	11:03:35	Yes
2	-0.003	-0.003	-0.002	11:03:39	Yes
3	-0.001	-0.001	-0.001	11:03:44	Yes
4	-0.002	-0.002	-0.001	11:03:49	Yes
5	-0.004	-0.004	-0.002	11:03:54	Yes
6	-0.003	-0.003	-0.002	11:03:58	Yes
7	-0.003	-0.003	-0.002	11:04:02	Yes
8	-0.004	-0.004	-0.002	11:04:07	Yes
9	-0.004	-0.004	-0.002	11:04:11	Yes
10	-0.004	-0.004	-0.002	11:04:15	Yes
Mean:	-0.003	-0.003	-0.002		
SD:	0.001	0.001	0.0007		
%RSD:	37.46	37.46	37.47		

==

Sequence No.: 10
Sample ID: SQC 2 ppm
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:04:37 AM
Data Type: Original

Replicate Data: SQC 2 ppm

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	1.996	1.996	0.781	11:04:38	Yes
2	1.980	1.980	0.777	11:04:43	Yes
3	1.999	1.999	0.782	11:04:47	Yes
4	1.989	1.989	0.779	11:04:51	Yes
5	1.998	1.998	0.781	11:04:55	Yes
6	2.001	2.001	0.782	11:05:00	Yes
7	1.994	1.994	0.780	11:05:04	Yes

===

Reprocessing Begun
Logged In Analyst: UM Jabatan Kimia Technique: AA Flame

Results Data Set (original): Mizi
Results Library (original): C:\data-AA\UM Jabatan Kimia\Results\Results.mdb
Results Data Set (reprocessed):
Results Library (reprocessed):

===

Method Loaded
Method Name: Pb Detection Method Last Saved: 3/24/2012 11:52:14 AM
Method Description: Pb Detection in eff

===

Sequence No.: 1 Autosampler Location: 1
Sample ID: blk Date Collected: 3/24/2012 2:01:00 PM
Analyst: Data Type: Reprocessed on 3/24/2012 3:08:54 PM
Logged In Analyst (Original) :

Replicate Data: blk

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[0.00]	0.025	14:01:04	No
2		[0.00]	0.025	14:01:08	No
3		[0.00]	0.025	14:01:12	No
Mean:		[0.00]	0.025		
SD:		0.00	0.0001		
%RSD:		0.00	0.23		

Auto-zero performed.

===

Sequence No.: 2 Autosampler Location: 2
Sample ID: 2 Date Collected: 3/24/2012 2:01:27 PM
Analyst: Data Type: Reprocessed on 3/24/2012 3:08:54 PM
Logged In Analyst (Original) :

Replicate Data: 2

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[2]	0.049	14:01:27	No
2		[2]	0.049	14:01:32	No
3		[2]	0.049	14:01:36	No
Mean:		[2]	0.049		
SD:		0	0.0003		
%RSD:		0	0.66		

Standard number 1 applied. [2]
Correlation Coef.: 1.000000 Slope: 0.02446 Intercept: 0.00000

===

Sequence No.: 3 Autosampler Location: 3
Sample ID: 4 Date Collected: 3/24/2012 2:02:06 PM
Analyst: Data Type: Reprocessed on 3/24/2012 3:08:54 PM
Logged In Analyst (Original) :

Replicate Data: 4

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[4]	0.098	14:02:09	No
2		[4]	0.098	14:02:13	No
3		[4]	0.099	14:02:18	No
Mean:		[4]	0.098		
SD:		0	0.0005		
%RSD:		0	0.52		

Standard number 2 applied. [4]
Correlation Coef.: 0.999999 Slope: 0.02448 Intercept: 0.00000

===

Sequence No.: 4 Autosampler Location: 4
Sample ID: 8 Date Collected: 3/24/2012 2:02:37 PM

Analyst: Data Type: Reprocessed on 3/24/2012 3:08:54 PM
Logged In Analyst (Original) :

Replicate Data: 8

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[8]	0.192	14:02:37	No
2		[8]	0.192	14:02:41	No
3		[8]	0.193	14:02:45	No
Mean:		[8]	0.192		
SD:		0	0.0004		
%RSD:		0	0.22		

Standard number 3 applied. [8]
Correlation Coef.: 0.999853   Slope: 0.02413   Intercept: 0.00000

Calibration data for Pb 283.31   Equation: Linear Through Zero

ID	Mean Signal (Abs)	Entered Conc. mg/L	Calculated Conc. mg/L	Standard Deviation	%RSD
blk	0.0000	0	0.000	0.00	0.2
2	0.0489	2.0	2.027	0.00	0.7
4	0.0979	4.0	4.059	0.00	0.5
8	0.1922	8.0	7.963	0.00	0.2

Correlation Coef.: 0.999853   Slope: 0.02413   Intercept: 0.00000

Sequence No.: 5   Autosampler Location:
Sample ID: 5ppm   Date Collected: 3/24/2012 2:04:14 PM
Analyst: Data Type: Reprocessed on 3/24/2012 3:08:54 PM
Logged In Analyst (Original) :

Replicate Data: 5ppm

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	4.678	4.678	0.113	14:04:16	No
2	4.668	4.668	0.113	14:04:20	No
3	4.634	4.634	0.112	14:04:25	No
Mean:	4.660	4.660	0.112		
SD:	0.023	0.023	0.0006		
%RSD:	0.492	0.492	0.49		

Sequence No.: 6   Autosampler Location:
Sample ID: Sample A   Date Collected: 3/24/2012 2:04:51 PM
Analyst: Data Type: Reprocessed on 3/24/2012 3:08:54 PM
Logged In Analyst (Original) :

Replicate Data: Sample A

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	-0.017	-0.017	-0.000	14:04:52	No
2	-0.013	-0.013	-0.000	14:04:57	No
3	-0.025	-0.025	-0.001	14:05:01	No
Mean:	-0.018	-0.018	-0.000		
SD:	0.006	0.006	0.0002		
%RSD:	35.04	35.04	35.04		

Sequence No.: 7   Autosampler Location:
Sample ID: Sample C   Date Collected: 3/24/2012 2:05:20 PM
Analyst: Data Type: Reprocessed on 3/24/2012 3:08:54 PM
Logged In Analyst (Original) :

Replicate Data: Sample C

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	0.049	0.049	0.001	14:05:20	No
2	0.049	0.049	0.001	14:05:24	No
3	0.048	0.048	0.001	14:05:28	No

Mean:	0.049	0.049	0.001
SD:	0.001	0.001	0.0000
%RSD:	1.122	1.122	1.12

==

Sequence No.: 8
Sample ID: Sample A spike 5 ppm
Analyst:
Logged In Analyst (Original) :

Autosampler Location:
Date Collected: 3/24/2012 2:10:17 PM
Data Type: Reprocessed on 3/24/2012 3:08:54 PM

Replicate Data: Sample A spike 5 ppm

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	5.071	5.071	0.122	14:10:20	No
2	5.056	5.056	0.122	14:10:24	No
3	5.030	5.030	0.121	14:10:29	No
Mean:	5.052	5.052	0.122		
SD:	0.021	0.021	0.0005		
%RSD:	0.416	0.416	0.42		

==

Sequence No.: 9
Sample ID: Sample C spike 3 ppm
Analyst:
Logged In Analyst (Original) :

Autosampler Location:
Date Collected: 3/24/2012 2:11:01 PM
Data Type: Reprocessed on 3/24/2012 3:08:54 PM

Replicate Data: Sample C spike 3 ppm

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	5.270	5.270	0.127	14:11:02	No
2	5.229	5.229	0.126	14:11:07	No
3	5.263	5.263	0.127	14:11:11	No
Mean:	5.254	5.254	0.127		
SD:	0.022	0.022	0.0005		
%RSD:	0.422	0.422	0.42		

==

Sequence No.: 10
Sample ID: 5ppm
Analyst:
Logged In Analyst (Original) :

Autosampler Location:
Date Collected: 3/24/2012 2:07:15 PM
Data Type: Reprocessed on 3/24/2012 3:08:54 PM

Replicate Data: 5ppm

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	4.966	4.966	0.120	14:07:17	No
2	4.995	4.995	0.121	14:07:21	No
3	4.972	4.972	0.120	14:07:25	No
Mean:	4.978	4.978	0.120		
SD:	0.016	0.016	0.0004		
%RSD:	0.314	0.314	0.31		

==

Method Loaded
Method Name: Fe in Effluent **Method Last Saved: 5/18/2013 11:43:53 AM**
Method Description: Fe in Effluent

==

Sequence No.: 20 **Autosampler Location:**
Sample ID: Blank **Date Collected: 5/18/2013 11:46:29 AM**
Analyst: **Data Type: Original**

Replicate Data: Blank

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[0.00]	0.163	11:46:29	Yes
2		[0.00]	0.164	11:46:34	Yes
3		[0.00]	0.164	11:46:38	Yes
4		[0.00]	0.164	11:46:42	Yes
5		[0.00]	0.164	11:46:47	Yes
6		[0.00]	0.163	11:46:51	Yes
7		[0.00]	0.164	11:46:55	Yes
8		[0.00]	0.164	11:47:00	Yes
9		[0.00]	0.164	11:47:04	Yes
10		[0.00]	0.163	11:47:08	Yes
Mean:		[0.00]	0.164		
SD:		0.00	0.0002		
%RSD:		0.00	0.15		

Auto-zero performed.

==

Sequence No.: 21 **Autosampler Location:**
Sample ID: Calib Std 1 **Date Collected: 5/18/2013 11:47:30 AM**
Analyst: **Data Type: Original**

Replicate Data: Calib Std 1

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[0.5]	0.049	11:47:31	Yes
2		[0.5]	0.050	11:47:36	Yes
3		[0.5]	0.049	11:47:40	Yes
4		[0.5]	0.049	11:47:44	Yes
5		[0.5]	0.050	11:47:49	Yes
6		[0.5]	0.051	11:47:53	Yes
7		[0.5]	0.051	11:47:57	Yes
8		[0.5]	0.053	11:48:02	Yes
9		[0.5]	0.053	11:48:06	Yes
10		[0.5]	0.052	11:48:10	Yes
Mean:		[0.5]	0.051		
SD:		0.0	0.0014		
%RSD:		0.0	2.70		

Standard number 1 applied. [0.5]
Correlation Coef.: 1.000000 Slope: 0.10139 Intercept: 0.00000

==

Sequence No.: 22 **Autosampler Location:**
Sample ID: Calib Std 2 **Date Collected: 5/18/2013 11:48:27 AM**
Analyst: **Data Type: Original**

Replicate Data: Calib Std 2

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[1]	0.116	11:48:29	Yes
2		[1]	0.115	11:48:33	Yes
3		[1]	0.116	11:48:38	Yes

4		[1]	0.116	11:48:42	Yes
5		[1]	0.115	11:48:46	Yes
6		[1]	0.115	11:48:51	Yes
7		[1]	0.116	11:48:55	Yes
8		[1]	0.115	11:48:59	Yes
9		[1]	0.115	11:49:04	Yes
10		[1]	0.115	11:49:08	Yes
Mean:		[1]	0.115		
SD:		0	0.0006		
%RSD:		0	0.50		

Standard number 2 applied. [1]
Correlation Coef.: 1.000000 Slope: 0.09040 Intercept: 0.00000

==

Sequence No.: 23
Sample ID: Calib Std 3
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:49:26 AM
Data Type: Original

Replicate Data: Calib Std 3

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[3]	0.323	11:49:27	Yes
2		[3]	0.325	11:49:31	Yes
3		[3]	0.324	11:49:36	Yes
4		[3]	0.325	11:49:40	Yes
5		[3]	0.324	11:49:44	Yes
6		[3]	0.321	11:49:49	Yes
7		[3]	0.324	11:49:53	Yes
8		[3]	0.326	11:49:57	Yes
9		[3]	0.324	11:50:02	Yes
10		[3]	0.325	11:50:06	Yes
Mean:		[3]	0.324		
SD:		0	0.0014		
%RSD:		0	0.42		

Standard number 3 applied. [3]
Correlation Coef.: 0.999001 Slope: 0.10757 Intercept: 0.00000
S-shaped calibration curve detected. Two-coefficient equation used.

Calibration data for Fe 248.33 **Equation: Nonlinear Through Zero**

ID	Mean Signal (Abs)	Entered Conc. mg/L	Calculated Conc. mg/L	Standard Deviation	%RSD
Blank	0.0000	0	0.000	0.00	0.2
Calib Std 1	0.0507	0.5	0.470	0.00	2.7
Calib Std 2	0.1154	1.0	1.066	0.00	0.5
Calib Std 3	0.3242	3.0	2.958	0.00	0.4

Correlation Coef.: 0.999001 Slope: 0.10757 Intercept: 0.00000

==

Sequence No.: 24
Sample ID: SQC 2 ppm
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:50:41 AM
Data Type: Original

Replicate Data: SQC 2 ppm

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	2.087	2.087	0.227	11:50:42	Yes
2	2.087	2.087	0.227	11:50:46	Yes
3	2.082	2.082	0.227	11:50:51	Yes
4	2.099	2.099	0.229	11:50:55	Yes
5	2.098	2.098	0.229	11:50:59	Yes
6	2.108	2.108	0.230	11:51:04	Yes
7	2.102	2.102	0.229	11:51:08	Yes
8	2.099	2.099	0.229	11:51:12	Yes

9	2.114	2.114	0.230	11:51:17	Yes
10	2.090	2.090	0.228	11:51:21	Yes
Mean:	2.097	2.097	0.229		
SD:	0.010	0.010	0.0011		
%RSD:	0.485	0.485	0.49		

===

Sequence No.: 25
Sample ID: Sample A
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:51:52 AM
Data Type: Original

Replicate Data: Sample A

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	-0.048	-0.048	-0.005	11:51:54	Yes
2	-0.054	-0.054	-0.006	11:51:58	Yes
3	-0.053	-0.053	-0.006	11:52:02	Yes
4	-0.053	-0.053	-0.006	11:52:06	Yes
5	-0.052	-0.052	-0.006	11:52:11	Yes
6	-0.052	-0.052	-0.006	11:52:15	Yes
7	-0.050	-0.050	-0.005	11:52:19	Yes
8	-0.054	-0.054	-0.006	11:52:24	Yes
9	-0.051	-0.051	-0.005	11:52:28	Yes
10	-0.054	-0.054	-0.006	11:52:32	Yes
Mean:	-0.052	-0.052	-0.006		
SD:	0.002	0.002	0.0002		
%RSD:	3.924	3.924	3.92		

===

Sequence No.: 26
Sample ID: Sample C
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:52:54 AM
Data Type: Original

Replicate Data: Sample C

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	-0.011	-0.011	-0.001	11:52:56	Yes
2	-0.015	-0.015	-0.002	11:53:00	Yes
3	-0.007	-0.007	-0.001	11:53:05	Yes
4	0.001	0.001	0.000	11:53:09	Yes
5	-0.002	-0.002	-0.000	11:53:14	Yes
6	-0.002	-0.002	-0.000	11:53:18	Yes
7	-0.005	-0.005	-0.001	11:53:22	Yes
8	0.000	0.000	-0.000	11:53:26	Yes
9	0.002	0.002	0.000	11:53:31	Yes
10	-0.006	-0.006	-0.001	11:53:35	Yes
Mean:	-0.005	-0.005	-0.000		
SD:	0.005	0.005	0.0006		
%RSD:	120.2	120.2	120.16		

===

Sequence No.: 27
Sample ID: SQC 2 ppm
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:54:07 AM
Data Type: Original

Replicate Data: SQC 2 ppm

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	2.027	2.027	0.221	11:54:09	Yes
2	2.022	2.022	0.220	11:54:13	Yes
3	2.075	2.075	0.226	11:54:17	Yes
4	2.075	2.075	0.226	11:54:22	Yes
5	2.029	2.029	0.221	11:54:26	Yes
6	2.085	2.085	0.227	11:54:30	Yes

7	2.050	2.050	0.223	11:54:35	Yes
8	2.069	2.069	0.225	11:54:39	Yes
9	2.062	2.062	0.225	11:54:43	Yes
10	2.073	2.073	0.226	11:54:48	Yes
Mean:	2.057	2.057	0.224		
SD:	0.023	0.023	0.0025		
%RSD:	1.122	1.122	1.14		

==

Method Loaded
Method Name: Ni in Effluent
Method Description: Ni in Effluen
Method Last Saved: 5/18/2013 11:21:59 AM

==

Sequence No.: 12
Sample ID: Blank
Analyst:
Autosampler Location:
Date Collected: 5/18/2013 11:26:34 AM
Data Type: Original

Replicate Data: Blank

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[0.00]	0.187	11:26:34	Yes
2		[0.00]	0.186	11:26:39	Yes
3		[0.00]	0.185	11:26:43	Yes
4		[0.00]	0.186	11:26:47	Yes
5		[0.00]	0.186	11:26:52	Yes
6		[0.00]	0.186	11:26:56	Yes
7		[0.00]	0.187	11:27:00	Yes
8		[0.00]	0.186	11:27:05	Yes
9		[0.00]	0.186	11:27:09	Yes
10		[0.00]	0.187	11:27:13	Yes
Mean:		[0.00]	0.186		
SD:		0.00	0.0005		
%RSD:		0.00	0.29		

Auto-zero performed.

==

Sequence No.: 13
Sample ID: Calib Std 1
Analyst:
Autosampler Location:
Date Collected: 5/18/2013 11:27:32 AM
Data Type: Original

Replicate Data: Calib Std 1

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[0.5]	0.053	11:27:33	Yes
2		[0.5]	0.055	11:27:37	Yes
3		[0.5]	0.054	11:27:42	Yes
4		[0.5]	0.051	11:27:46	Yes
5		[0.5]	0.052	11:27:50	Yes
6		[0.5]	0.053	11:27:54	Yes
7		[0.5]	0.053	11:27:58	Yes
8		[0.5]	0.053	11:28:03	Yes
9		[0.5]	0.052	11:28:07	Yes
10		[0.5]	0.053	11:28:11	Yes
Mean:		[0.5]	0.053		
SD:		0.0	0.0009		
%RSD:		0.0	1.75		

Standard number 1 applied. [0.5]
Correlation Coef.: 1.000000 Slope: 0.10566 Intercept: 0.00000

==

Sequence No.: 14
Sample ID: Calib Std 2
Analyst:
Autosampler Location:
Date Collected: 5/18/2013 11:28:31 AM
Data Type: Original

Replicate Data: Calib Std 2

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[1]	0.120	11:28:32	Yes
2		[1]	0.119	11:28:36	Yes
3		[1]	0.120	11:28:41	Yes

4	[1]	0.121	11:28:45	Yes
5	[1]	0.119	11:28:49	Yes
6	[1]	0.120	11:28:54	Yes
7	[1]	0.120	11:28:58	Yes
8	[1]	0.119	11:29:03	Yes
9	[1]	0.119	11:29:07	Yes
10	[1]	0.119	11:29:11	Yes
Mean:	[1]	0.119		
SD:	0	0.0007		
%RSD:	0	0.58		

Standard number 2 applied. [1]
Correlation Coef.: 1.000000 Slope: 0.09470 Intercept: 0.00000

==

Sequence No.: 15
Sample ID: Calib Std 3
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:29:31 AM
Data Type: Original

Replicate Data: Calib Std 3

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[3]	0.316	11:29:33	Yes
2		[3]	0.319	11:29:37	Yes
3		[3]	0.318	11:29:41	Yes
4		[3]	0.316	11:29:46	Yes
5		[3]	0.318	11:29:50	Yes
6		[3]	0.319	11:29:54	Yes
7		[3]	0.319	11:29:59	Yes
8		[3]	0.318	11:30:03	Yes
9		[3]	0.317	11:30:07	Yes
10		[3]	0.318	11:30:11	Yes
Mean:		[3]	0.318		
SD:		0	0.0011		
%RSD:		0	0.35		

Standard number 3 applied. [3]
Correlation Coef.: 0.998777 Slope: 0.11385 Intercept: 0.00000
S-shaped calibration curve detected. Two-coefficient equation used.

Calibration data for Ni 232.00 **Equation: Nonlinear Through Zero**

ID	Mean Signal (Abs)	Entered Conc. mg/L	Calculated Conc. mg/L	Standard Deviation	%RSD
Blank	0.0000	0	0.000	0.00	0.3
Calib Std 1	0.0528	0.5	0.468	0.00	1.8
Calib Std 2	0.1195	1.0	1.071	0.00	0.6
Calib Std 3	0.3178	3.0	2.950	0.00	0.4

Correlation Coef.: 0.998777 Slope: 0.11385 Intercept: 0.00000

==

Sequence No.: 16
Sample ID: SQC 2 ppm
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:30:43 AM
Data Type: Original

Replicate Data: SQC 2 ppm

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	2.040	2.040	0.223	11:30:44	Yes
2	2.051	2.051	0.225	11:30:49	Yes
3	2.030	2.030	0.222	11:30:53	Yes
4	2.038	2.038	0.223	11:30:57	Yes
5	2.037	2.037	0.223	11:31:02	Yes
6	2.040	2.040	0.223	11:31:06	Yes
7	2.031	2.031	0.223	11:31:10	Yes
8	2.037	2.037	0.223	11:31:15	Yes

9	2.033	2.033	0.223	11:31:19	Yes
10	2.042	2.042	0.224	11:31:23	Yes
Mean:	2.038	2.038	0.223		
SD:	0.006	0.006	0.0006		
%RSD:	0.299	0.299	0.29		

==

Sequence No.: 17
Sample ID: Sample A
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:31:47 AM
Data Type: Original

Replicate Data: Sample A

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	-0.022	-0.022	-0.003	11:31:48	Yes
2	-0.032	-0.032	-0.004	11:31:52	Yes
3	-0.049	-0.049	-0.006	11:31:57	Yes
4	-0.041	-0.041	-0.005	11:32:01	Yes
5	-0.034	-0.034	-0.004	11:32:06	Yes
6	-0.049	-0.049	-0.006	11:32:10	Yes
7	-0.050	-0.050	-0.006	11:32:15	Yes
8	-0.040	-0.040	-0.005	11:32:19	Yes
9	-0.042	-0.042	-0.005	11:32:23	Yes
10	-0.038	-0.038	-0.004	11:32:28	Yes
Mean:	-0.040	-0.040	-0.005		
SD:	0.009	0.009	0.0010		
%RSD:	21.59	21.59	21.60		

==

Sequence No.: 18
Sample ID: Sample C
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:32:51 AM
Data Type: Original

Replicate Data: Sample C

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	0.011	0.011	0.001	11:32:53	Yes
2	-0.008	-0.008	-0.001	11:32:57	Yes
3	0.005	0.005	0.001	11:33:01	Yes
4	0.013	0.013	0.001	11:33:06	Yes
5	0.009	0.009	0.001	11:33:10	Yes
6	0.013	0.013	0.001	11:33:14	Yes
7	0.003	0.003	0.000	11:33:19	Yes
8	0.003	0.003	0.000	11:33:23	Yes
9	0.012	0.012	0.001	11:33:27	Yes
10	0.004	0.004	0.000	11:33:32	Yes
Mean:	0.006	0.006	0.001		
SD:	0.007	0.007	0.0008		
%RSD:	103.9	103.9	103.88		

==

Sequence No.: 19
Sample ID: SQC 2 ppm
Analyst:

Autosampler Location:
Date Collected: 5/18/2013 11:34:07 AM
Data Type: Original

Replicate Data: SQC 2 ppm

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	2.078	2.078	0.228	11:34:08	Yes
2	2.066	2.066	0.226	11:34:13	Yes
3	2.061	2.061	0.226	11:34:17	Yes
4	2.057	2.057	0.225	11:34:21	Yes
5	2.058	2.058	0.225	11:34:26	Yes
6	2.061	2.061	0.226	11:34:30	Yes

7	2.057	2.057	0.225	11:34:34	Yes
8	2.079	2.079	0.228	11:34:39	Yes
9	2.049	2.049	0.224	11:34:43	Yes
10	2.049	2.049	0.224	11:34:47	Yes
Mean:	2.061	2.061	0.226		
SD:	0.010	0.010	0.0011		
%RSD:	0.505	0.505	0.49		

Analysis Begun

Logged In Analyst: UM Jabatan Kimia   Technique: AA Flame
Spectrometer Model: AAnalyst 400, S/N 201S7081502   Autosampler Model:

Sample Information File: C:\data-AA\UM Jabatan Kimia\Sample Information\Untitled.SIF
Batch ID:
Results Data Set:
Results Library:

Method Loaded
Method Name: Cu   Method Last Saved: 5/11/2013 11:00:19 AM
Method Description: Cu detection in Waste Water

Sequence No.: 23   Autosampler Location:
Sample ID: Blank   Date Collected: 5/11/2013 11:43:49 AM
Analyst: Data Type: Original

Replicate Data: Blank

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[0.00]	0.001	11:43:50	No
2		[0.00]	0.001	11:43:55	No
3		[0.00]	0.002	11:43:59	No
4		[0.00]	0.000	11:44:03	No
5		[0.00]	0.003	11:44:07	No
6		[0.00]	0.000	11:44:12	No
7		[0.00]	-0.000	11:44:16	No
8		[0.00]	0.003	11:44:21	No
9		[0.00]	-0.000	11:44:25	No
10		[0.00]	-0.000	11:44:29	No
Mean:		[0.00]	0.001		
SD:		0.00	0.0013		
%RSD:		0.00	134.70		

Auto-zero performed.

Sequence No.: 24   Autosampler Location:
Sample ID: Std 1   Date Collected: 5/11/2013 11:44:48 AM
Analyst: Data Type: Original

Replicate Data: Std 1

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[0.4]	0.064	11:44:49	No
2		[0.4]	0.064	11:44:54	No
3		[0.4]	0.065	11:44:58	No
4		[0.4]	0.065	11:45:02	No
5		[0.4]	0.067	11:45:07	No
6		[0.4]	0.067	11:45:11	No
7		[0.4]	0.067	11:45:15	No
8		[0.4]	0.069	11:45:20	No
9		[0.4]	0.068	11:45:24	No
10		[0.4]	0.067	11:45:28	No
Mean:		[0.4]	0.066		
SD:		0.0	0.0017		
%RSD:		0.0	2.56		

Standard number 1 applied. [0.4]
Correlation Coef.: 0.997234   Slope: 0.19761   Intercept: 0.00000

Sequence No.: 25   Autosampler Location:
Sample ID: Std 2   Date Collected: 5/11/2013 11:45:52 AM
Analyst: Data Type: Original

Replicate Data: Std 2

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[1]	0.197	11:45:53	No
2		[1]	0.200	11:45:57	No
3		[1]	0.202	11:46:01	No
4		[1]	0.202	11:46:06	No
5		[1]	0.204	11:46:10	No
6		[1]	0.205	11:46:14	No
7		[1]	0.205	11:46:19	No
8		[1]	0.205	11:46:23	No
9		[1]	0.205	11:46:27	No
10		[1]	0.205	11:46:32	No
Mean:		[1]	0.203		
SD:		0	0.0028		
%RSD:		0	1.37		

Standard number 2 applied. [1]
Correlation Coef.: 0.996629 Slope: 0.19901 Intercept: 0.00000

==

Sequence No.: 26
Sample ID: Std 3
Analyst:

Autosampler Location:
Date Collected: 5/11/2013 11:47:01 AM
Data Type: Original

Replicate Data: Std 3

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1		[1.6]	0.318	11:47:02	No
2		[1.6]	0.316	11:47:07	No
3		[1.6]	0.318	11:47:11	No
4		[1.6]	0.317	11:47:15	No
5		[1.6]	0.318	11:47:20	No
6		[1.6]	0.319	11:47:24	No
7		[1.6]	0.319	11:47:29	No
8		[1.6]	0.315	11:47:33	No
9		[1.6]	0.318	11:47:37	No
10		[1.6]	0.318	11:47:42	No
Mean:		[1.6]	0.318		
SD:		0.0	0.0012		
%RSD:		0.0	0.38		

Standard number 3 applied. [1.6]
Correlation Coef.: 0.996633 Slope: 0.19858 Intercept: 0.00000
The calibration curve may not be linear.

Calibration data for Cu 324.75 **Equation: Linear Through Zero**

ID	Mean Signal (Abs)	Entered Conc. mg/L	Calculated Conc. mg/L	Standard Deviation	%RSD
Blank	0.0000	0	0.000	0.00	134.7
Std 1	0.0663	0.4	0.334	0.00	2.6
Std 2	0.2029	1.0	1.022	0.00	1.4
Std 3	0.3177	1.6	1.600	0.00	0.4

Correlation Coef.: 0.996633 Slope: 0.19858 Intercept: 0.00000

==

Sequence No.: 27
Sample ID: SQC
Analyst:

Autosampler Location:
Date Collected: 5/11/2013 11:48:07 AM
Data Type: Original

Replicate Data: SQC

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	1.278	1.278	0.254	11:48:08	No
2	1.277	1.277	0.254	11:48:13	No
3	1.275	1.275	0.253	11:48:17	No
4	1.274	1.274	0.253	11:48:21	No
5	1.281	1.281	0.254	11:48:26	No
6	1.283	1.283	0.255	11:48:30	No
7	1.284	1.284	0.255	11:48:34	No

8	1.286	1.286	0.255	11:48:39	No
9	1.272	1.272	0.253	11:48:43	No
10	1.272	1.272	0.253	11:48:48	No
Mean:	1.278	1.278	0.254		
SD:	0.005	0.005	0.0010		
%RSD:	0.390	0.390	0.39		

==

Sequence No.: 28
Sample ID: Sample A
Analyst:

Autosampler Location:
Date Collected: 5/11/2013 11:49:13 AM
Data Type: Original

Replicate Data: Sample A

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	-0.001	-0.001	-0.000	11:49:15	No
2	0.002	0.002	0.000	11:49:19	No
3	-0.004	-0.004	-0.001	11:49:24	No
4	0.002	0.002	0.000	11:49:28	No
5	0.000	0.000	-0.000	11:49:32	No
6	-0.003	-0.003	-0.001	11:49:37	No
7	-0.001	-0.001	-0.000	11:49:41	No
8	-0.003	-0.003	-0.001	11:49:45	No
9	-0.001	-0.001	-0.000	11:49:50	No
10	-0.004	-0.004	-0.001	11:49:54	No
Mean:	-0.001	-0.001	-0.000		
SD:	0.002	0.002	0.0004		
%RSD:	180.5	180.5	180.47		

==

Sequence No.: 29
Sample ID: Sample C
Analyst:

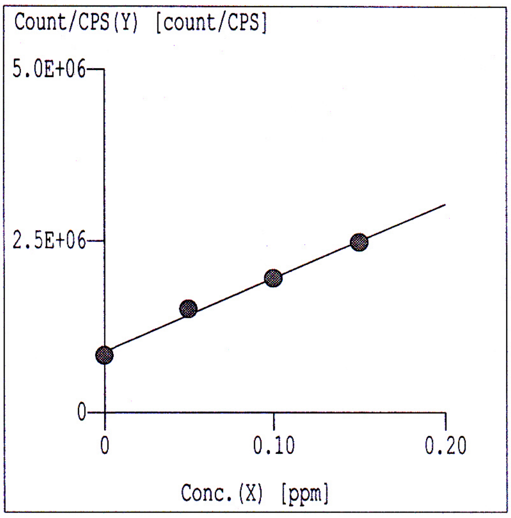
Autosampler Location:
Date Collected: 5/11/2013 11:50:16 AM
Data Type: Original

Replicate Data: Sample C

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Time	Signal Stored
1	0.004	0.004	0.001	11:50:18	No
2	0.003	0.003	0.001	11:50:22	No
3	0.003	0.003	0.001	11:50:27	No
4	0.004	0.004	0.001	11:50:31	No
5	0.004	0.004	0.001	11:50:35	No
6	0.004	0.004	0.001	11:50:40	No
7	0.005	0.005	0.001	11:50:44	No
8	0.004	0.004	0.001	11:50:48	No
9	0.005	0.005	0.001	11:50:53	No
10	0.004	0.004	0.001	11:50:57	No
Mean:	0.004	0.004	0.001		
SD:	0.000	0.000	0.0001		
%RSD:	11.94	11.94	11.94		

=== Graph Detail ===

Step	Mass	Element	ISTD	Unit
(2)	56	Fe	---	ppm

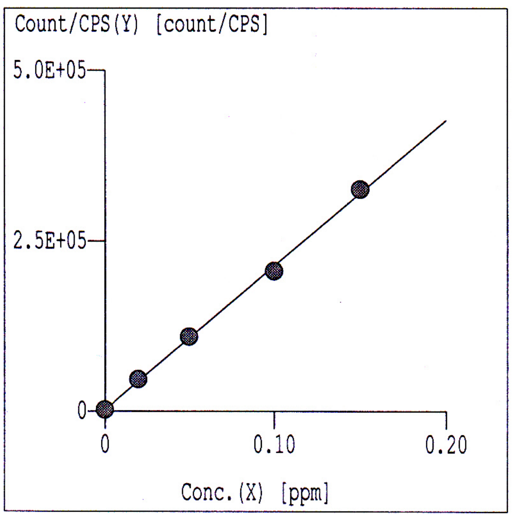


	Rjct	Conc	Calc Conc	CPS/Count	Ratio		RSD [%]
1		0.000	-5.067E-03	8.244E+05	---	P	---
2	ON	2.000E-02	---	1.438E+06	---	A	---
3		5.000E-02	5.793E-02	1.504E+06	---	A	---
4		1.000E-01	9.935E-02	1.952E+06	---	A	---
5		1.500E-01	1.478E-01	2.475E+06	---	A	---
6		---	---	---	---		---
7		---	---	---	---		---
8		---	---	---	---		---
9		---	---	---	---		---
10		---	---	---	---		---
11		---	---	---	---		---
12		---	---	---	---		---
13		---	---	---	---		---
14		---	---	---	---		---
15		---	---	---	---		---
16		---	---	---	---		---
17		---	---	---	---		---
18		---	---	---	---		---
19		---	---	---	---		---
20		---	---	---	---		---

Curve Fit: Y=aX+b
r = 0.9963
Y = 1.080E+007*X +8.791E+005
X = 9.264E-008*Y -8.144E-002
DL = --- ppm
BEC = 8.144E-02 ppm

Weight: OFF
Min Conc: 0.000

Step	Mass	Element	ISTD	Unit
(2)	60	Ni	---	ppm



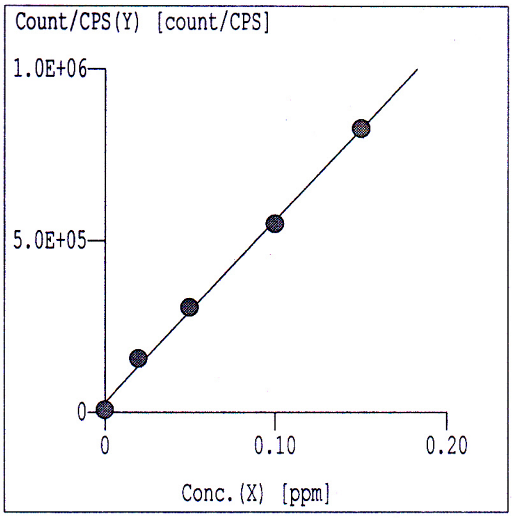
	Rjct	Conc	Calc Conc	CPS/Count	Ratio		RSD [%]
1		0.000	-4.909E-05	1666	---	P	---
2		2.000E-02	2.104E-02	4.649E+04	---	P	---
3		5.000E-02	5.069E-02	1.095E+05	---	P	---
4		1.000E-01	9.604E-02	2.059E+05	---	P	---
5		1.500E-01	1.523E-01	3.254E+05	---	P	---
6		---	---	---	---		---
7		---	---	---	---		---
8		---	---	---	---		---
9		---	---	---	---		---
10		---	---	---	---		---
11		---	---	---	---		---
12		---	---	---	---		---
13		---	---	---	---		---
14		---	---	---	---		---
15		---	---	---	---		---
16		---	---	---	---		---
17		---	---	---	---		---
18		---	---	---	---		---
19		---	---	---	---		---
20		---	---	---	---		---

Curve Fit: Y=aX+b
r = 0.9993
Y = 2.125E+006*X +1.771E+003
X = 4.706E-007*Y -8.332E-004
DL = --- ppm
BEC = 8.332E-04 ppm

Weight: OFF
Min Conc: 0.000

=== Graph Detail ===

Step Mass Element ISTD Unit
(2) 63 Cu --- ppm

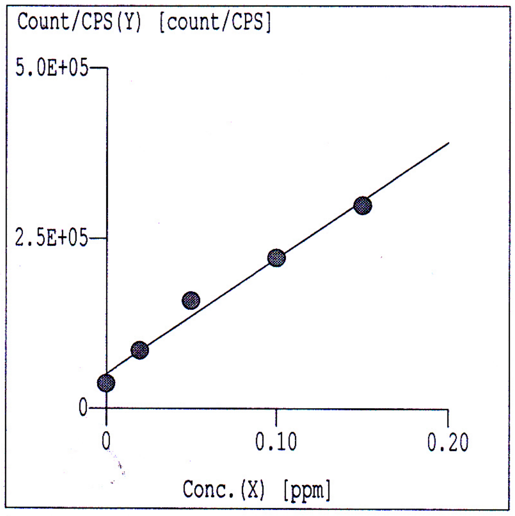


	Rjct	Conc	Calc Conc	CPS/Count	Ratio		RSD [%]
1		0.000	-4.006E-03	6968	---	P	---
2		2.000E-02	2.393E-02	1.557E+05	---	P	---
3		5.000E-02	5.197E-02	3.049E+05	---	P	---
4		1.000E-01	9.786E-02	5.491E+05	---	P	---
5		1.500E-01	1.502E-01	8.280E+05	---	P	---
6		---	---	---	---		---
7		---	---	---	---		---
8		---	---	---	---		---
9		---	---	---	---		---
10		---	---	---	---		---
11		---	---	---	---		---
12		---	---	---	---		---
13		---	---	---	---		---
14		---	---	---	---		---
15		---	---	---	---		---
16		---	---	---	---		---
17		---	---	---	---		---
18		---	---	---	---		---
19		---	---	---	---		---
20		---	---	---	---		---

Curve Fit: Y=aX+b
r = 0.9987
Y = 5.322E+006*X +2.829E+004
X = 1.879E-007*Y -5.316E-003
DL = --- ppm
BEC = 5.316E-03 ppm

Weight: OFF
Min Conc: 0.000

Step Mass Element ISTD Unit
(2) 66 Zn --- ppm



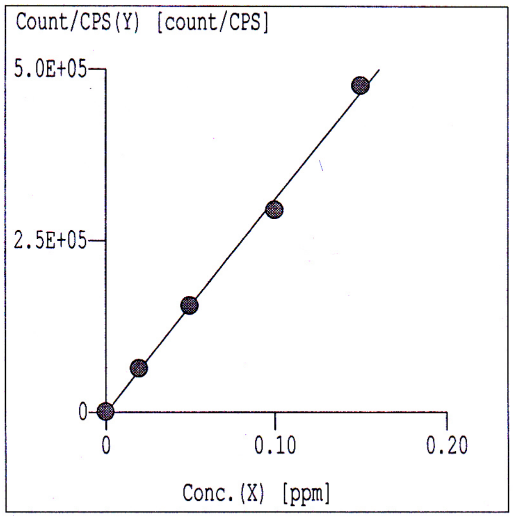
	Rjct	Conc	Calc Conc	CPS/Count	Ratio		RSD [%]
1		0.000	-8.725E-03	3.621E+04	---	P	---
2		2.000E-02	1.986E-02	8.476E+04	---	P	---
3		5.000E-02	6.319E-02	1.584E+05	---	P	---
4		1.000E-01	1.002E-01	2.211E+05	---	P	---
5		1.500E-01	1.455E-01	2.982E+05	---	P	---
6		---	---	---	---		---
7		---	---	---	---		---
8		---	---	---	---		---
9		---	---	---	---		---
10		---	---	---	---		---
11		---	---	---	---		---
12		---	---	---	---		---
13		---	---	---	---		---
14		---	---	---	---		---
15		---	---	---	---		---
16		---	---	---	---		---
17		---	---	---	---		---
18		---	---	---	---		---
19		---	---	---	---		---
20		---	---	---	---		---

Curve Fit: Y=aX+b
r = 0.9911
Y = 1.698E+006*X +5.103E+004
X = 5.888E-007*Y -3.004E-002
DL = --- ppm
BEC = 3.004E-02 ppm

Weight: OFF
Min Conc: 0.000

=== Graph Detail ===

Step Mass Element ISTD Unit
(1) 111 Cd --- ppm

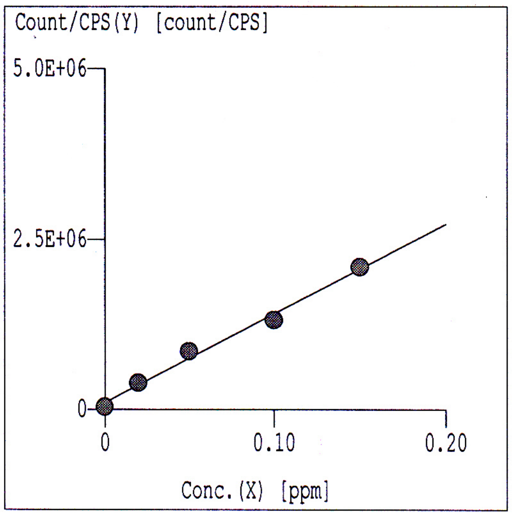


	Rjct	Conc	Calc Conc	CPS/Count	Ratio		RSD [%]
1		0.000	7.611E-04	518.0	---	P	---
2		2.000E-02	2.086E-02	6.328E+04	---	P	---
3		5.000E-02	5.024E-02	1.551E+05	---	P	---
4		1.000E-01	9.500E-02	2.949E+05	---	P	---
5		1.500E-01	1.531E-01	4.764E+05	---	P	---
6		---	---	---	---		---
7		---	---	---	---		---
8		---	---	---	---		---
9		---	---	---	---		---
10		---	---	---	---		---
11		---	---	---	---		---
12		---	---	---	---		---
13		---	---	---	---		---
14		---	---	---	---		---
15		---	---	---	---		---
16		---	---	---	---		---
17		---	---	---	---		---
18		---	---	---	---		---
19		---	---	---	---		---
20		---	---	---	---		---

Curve Fit: Y=aX+b
r = 0.9988
Y = 3.123E+006*X -1.859E+003
X = 3.202E-007*Y +5.953E-004
DL = --- ppm
BEC = -5.953E-04 ppm

Weight: OFF
Min Conc: 0.000

Step Mass Element ISTD Unit
(1) 208 Pb --- ppm



	Rjct	Conc	Calc Conc	CPS/Count	Ratio		RSD [%]
1		0.000	-4.556E-03	3.486E+04	---	P	---
2		2.000E-02	2.218E-02	3.866E+05	---	P	---
3		5.000E-02	5.760E-02	8.525E+05	---	P	---
4		1.000E-01	9.280E-02	1.316E+06	---	A	---
5		1.500E-01	1.520E-01	2.094E+06	---	A	---
6		---	---	---	---		---
7		---	---	---	---		---
8		---	---	---	---		---
9		---	---	---	---		---
10		---	---	---	---		---
11		---	---	---	---		---
12		---	---	---	---		---
13		---	---	---	---		---
14		---	---	---	---		---
15		---	---	---	---		---
16		---	---	---	---		---
17		---	---	---	---		---
18		---	---	---	---		---
19		---	---	---	---		---
20		---	---	---	---		---

Curve Fit: Y=aX+b
r = 0.9954
Y = 1.315E+007*X +9.479E+004
X = 7.602E-008*Y -7.206E-003
DL = --- ppm
BEC = 7.206E-03 ppm

Weight: OFF
Min Conc: 0.000

	Fe / 56 [#2]	Ni / 60 [#2]	Cu / 63 [#2]	Cd / 111 [#1]	Pb / 208 [#1]
d:\sahar\300513.B\006SMPL.D	0.12	0.12	0.12	0.13	0.12
d:\sahar\300513.B\007SMPL.D	-0.02	0.01	0.00	0.00	0.00
d:\sahar\300513.B\008SMPL.D	-0.04	0.00	0.00	0.00	-0.01
d:\sahar\300513.B\009SMPL.D	0.11	0.12	0.12	0.13	0.13