DETERMINATION OF LEAD, CADMIUM, ZINC AND NICKEL IN EDIBLE OIL PRODUCTS BY STRIPPING VOLTAMMETRY

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DISSERTATION SUBMITTED IN FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE IN ANALYTICAL CHEMISTRY AND INSTRUMENTATION

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ABSTRACT

Edible oil products had become an everyday food that been consumed by most of the people either for dressing or for cooking purpose. Metal has become a common contamination to the food product, which it found the way into the edible oil through environment and processing contamination such as crushing, distillation, and hydrogenation. In this Study, the main focuses of metal analysis are only confined to 4 common metals which are the Cadmium, Lead, Zinc and Nickel. These elements are selected in this study due to reason that Cadmium and Lead are commonly known for their contamination from environment and from processing equipment, and it is important to be analyzed due to its health deteriorations to consumer. Nickel was chosen in this analysis for its well known function as a catalyst for hydrogenation process in edible oil and Zinc was known to be a micronutrient however excess concentration maybe impact to human health. From analytical chemistry point of view, Spectroscopic techniques particularly Atomic Absorption Spectroscopy (AAS) and Inductively Couple Plasma Optical Emission Spectroscopy (ICP-OES) are some of the well known techniques used for the analysis of metal, however in this study a second approach by electrochemistry technique is employed to study and quantify the metal concentration, the method used is known as Voltammetry Stripping Technique. The advantages of voltammetry technique in comparison to others is it allows for detection limit of sub part per billion level, non destruction method as well as simultaneous determination of some metals. In this study, standard addition method was used as the calibration technique for the determination of Zinc, Nickel, Lead and Cadmium in the edible oil products.

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1.0 INTRODUCTION

Vegetable oil or vegetable fat is fall in same chemical group know as lipid that is extracted from plant sources. The different between vegetable fat and oil is basically in term of it morphological, the term oil is referring to vegetable that exist in liquid form at room temperature such as cooking oil, while vegetable fat are referring to solid at room temperature such as margarine. Vegetable fat and oil are triglyceride that is consist of a glycerol that attached to three molecule of fatty acid by an Ester bond, the chemical structure of triglyceride is show in figure below:

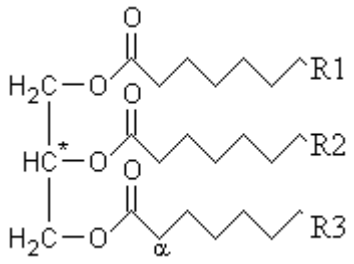


Figure 1.1 Chemical Structure of Triglyceride

The source of Vegatable oil and fat can be come from variety of plant for example it can be from Palm Oil, Sunflower Seed, Rape Seed, Olive, Soya bean, and Corn. The content of saturated and unsaturated fatty Acid can be varied among the source, and it is usually important as it determine the morphological properties as well as it application used in the industry.

Vegetable oil had become more important in our daily life; it is widely used not only in cooking and food processing (Edible Oil) but also in oleo-chemical which provide an ample application included biodiesel, pharmaceutical such as tocopherol (vitamin E), chemical industries and as well in cosmetic (such as glycerin). From the numerous application of the vegetable oil, Edible oil is of highly concern in term of it quality especially in heavy metal content as it had an implication toward health issue. The source of metal contamination can be come from many sources, in basic it can be divide into two types endogenous and exogenous (Pehlivan, Arslan, Gode and etc, 2008). Endogenous factor can be due to the metabolism of the plant it self which is depend on the soil, fertilizer and water where the plant growth. The metal take up by the plant is very depend on the amount and type of metal that content in the soil, water or fertilizer that used in the plant (Rehana, Tasneem and etc, 2009). In the cases of exogenous sources it can be causes by metal that contaminated the vegetable oil during agronomic technique of processing such as extracting, crushing, refining, bleaching, and hydrogenation (Pehlivan, Arslan, Gode and etc, 2008), for example Nickel (Ni) contamination can be causes by the hydrogenation process.

Ample of study and research paper had been publish in the analysis of heavy metal in edible oil, from the many research work the most common heavy metal analysis included Iron (Fe), Copper (Cu), Nickel (Ni), Zinc (Zn), Lead (Pb), Cadmium (Cd) and Chromium (Cr). This metal is of greatly interest in edible oil analysis because the metal are known to have a catalyst effect which accelerate the auto-oxidation mechanism that lead to oxidation or rancidity of the vegetable oil, especially Iron, Copper, and Nickel

(Mareila and Reinaldo, 2006). Other metal lead, Cadmium and chromium is of highly interest due to it toxicity effect. The main metals of interest in this study are basically focused on Zinc, Cadmium, Lead and Nickel in edible oil, this metal are selected because it is the most commonly assess for quality of the Edible oil

Nickel contain in Vegetable oil can be come from many sources, either from the process itself or from the process equipment. The hydrogenation is a very important process in vegetable oil especially in the production of margarine and shortening. The main objective of hydrogenation process is mainly to modify the oxidative and thermal stability properties of the edible oil which had an effect on the physical properties such as melting temperature, Surface tension, viscosity and etc (Maria, Gabriela Guillermo and etc, 2007). The hydrogenation processes are usually performed by adding nickel or copper as a catalyst to accelerate the process. The nickel is preferable in contrast to copper due to cost, as nickel is cheaper and in most cases copper is difficult to be removed from the process (M.K Gupta, 2005). Apart from that, nickel contamination in edible oil can also be causes by corrosion of the processing equipment (Mariela and Reinaldo, 2006).The permitted amount of nickel in oil is set by many national and international country at level of 0.2mg/Kg (Durali, Ozgur and etc, 2009).

Cadmium and lead contamination are hardly associated to edible oil processing, it is mostly are cause by the environment exposure rather than process related. It present can be causes by deposition or bioaccumulation from the soil or water due to pollution. Cadmium and Lead are know to be accumulate in biological system for long half life. the

source of cadmium and lead (Pb) can be due to combustion of fuel in refinery process, industrial emission, and from packaging material such as colorant and stabilizer in plastic (Giacomo, Lara and Et al). It is also know to be highly contaminated from phosphate base fertilizer (Rehana, Tasneem and etc, 2009) that used in the plantation. Lead and cadmium are of greatly interested in edible oil analysis because of it toxicity which had an implication to the health. Lead and cadmium are known to be acute and chronic poisoning which can causes failure to the kidney, liver, heart and immune system. In addition to that, exposure to such metal is also known to cause chromosome aberration, cancer, and birth defect. As according to Durali, ozgur and etc that many national and international countries had set the permitted amount of lead (Pb) in oil at 0.1mg/Kg and Cadmium (Cd) at 0.05 mg/Kg. As according to World Health Organization (WHO) the permitted doses of Cadmium (Cd) and lead (Pb) for an adult per week are set at 0.5 mg and 3.0 mg respectively.

Zinc are mineral that exist in edible oil due to it function as metabolite and micronutrient for the plant. The source of zinc are mainly from the soil which the plant growth and as well from the water. Zinc is an essential mineral that required by human body, which is important for normal growth, wound healing, normal taste sensational, and appetite. Zinc usually is required at low concentration; however at high concentration of Zinc it can pose some side effect that had health deleterious impact. The daily recommended amount of zinc (Zn) intake for an adult male is at 15 mg and for adult female is at 12 mg (Durali, Ozgur and etc, 2009).

2.0 OBJECTIVE OF STUDY

The objective with respect to the topic of 'Determination of lead, Cadmium, Nickel and Zinc in Edible Oil by Stripping Voltammetry' is to study the analysis method for quantify the concentration of the metal in the various edible oil sample by using electrochemistry instrument particular stripping Voltammetry technique. In this study the Lead, Cadmium and Zinc will be analyze by using the Anodic Stripping Voltammetry Technique, whereas for Nickel will be analyzed by using Adsorptive Stripping Voltammetry. In order to assess the method reliability a recovery study is perform on the spike sample for both stripping method.

3.0 PRINCIPLE OF ANALYSIS

The propose method in this work was focus on the analysis of Zinc, Cadmium, Lead and Nickel in Edible oil. Prior to metal analysis the sample are prepared by digestion using microwave digestion technique and the metal is analyze by using stripping voltammetry method, therefore in this chapter the principle of this analysis method will be highlighted for better understanding of the analysis method.

3.1 MICROWAVE DIGESTION

Microwave Digestion technique had been introduce since 1980, and it had become an important and preferable sample preparation technique especially in the

analysis of metal. Microwave digestion method is a simple and direct method, the principle of this method is basically based on microwave electromagnetic radiation that applied to the sample, as a consequences of this the energy of the microwave is absorb by the water molecule that present in the digested solution (sample), which subsequently causes the dipole rotation of the water molecule. Heat is produced as a result of friction of the molecule movement, which is then conducted within the solution. By this mechanism it heat up the solution of sample much faster in comparison to conventional wet digestion technique and the organic matrix of the sample can be breakdown or oxidized when suitable mineral acid is used.

A basic microwave digester instrument are consists of a microwave chamber, carousel, cooling fan, temperature sensor and sample vessel. The microwave chamber is where the sample carousel is put in place prior to digestion. A carousel usually is turn able which allow for sample to be homogenously irradiated in the microwave chamber. Cooling fan is basically functioning for cool down the sample upon completion of sample digestion. In most microwave digester device that available in the market, is usually equipped with temperature sensor which allow for accurate temperature control during digestion process, in some cases whereby pressure sensor is also included for pressure monitoring. In today advance microwave digester, almost of the system is able to be monitor by microcomputer either built inside the system or can be connected to computer directly, this allow for easy setting of the sample digestion condition such as microwave power (watt), ramp time, temperature, hold time, cooling time and as well the pressure setting, apart from that some software allow as well for recording of each sample vessel

digestion condition, sample identification, date, time and method used. The type of sample vessel used is an important consideration it often very depend on the characteristic of sample and mineral acid to be used. There are various types of sample vessel used in microwave digester such as Polytetraflouroethylene (PTFE), Quartz, Polyflouroacetate (PFA) and many more. The type of material used for the microwave sample vessel must fulfill certain requirement. One of the most important criteria of the sample vessel is that it must be transparent to the microwave so that the Electromagnetic radiation can direct interact with the sample solution, apart from that the vessel as well must be able to withstand the corrosiveness of mineral acid or oxidizing agent used such as concentrated nitric acid (65%) and hydrogen peroxide, and the vessel as well must able to withstand heat and pressure. The figure (3.1) below show an example of a microwave digester

Figure 3.1 Microwave Digester Systems

Microwave digester, is preferable by most of the analysis of metal in organic material due to the system is efficient, usually the sample digestion time can be greatly reduce and beside that the temperature and pressure can be controlled in comparison to conventional wet digestion method. One of the added advantages is that, the digestion is perform in closed vessel which is important to prevent any lost of analyte by evaporation and also contamination from environment especially when perform analysis at a trace level. Apart from that, the sample vessel is transparent to microwave as a result the sample vessel is not heated and can be easily handle after the sample digestion. However the disadvantage of microwave digester is that only a small amount of sample can be used which is in the range of 0.1-0.5 g. The amount of sample to be used is very depend on the carbon content in the sample, the principle guidelines is the carbon content should not exceed 1.5 mg Carbon per volume (ml) of the liner sample vessel (Pier Luigi Buldini, Loretta Ricci and Jawahar Sharma, 2002)

The used of type of mineral acid for sample digestion is very depend on the type of sample to be digest, some of the common mineral acid used for digestion included hydrochloric acid (HCl), Sulfuric Acid, Nitric Acid, Hydrofluoric Acid, Perchloric acid and many more. However the general guidelines for digestion of sample containing organic matrices such as food sample, Nitric acid are often used due to it oxidizing properties, therefore for the cases of edible oil, Nitric Acid is the choice of acid to be used. In microwave digestion the amount of sample is important as it affected the optimization of the sample dissolution. However, small sample amount during sample preparation may pose a difficulty in trace metal analysis, as a result the microwave

digestion method may causes limitation for used with analytical equipment such as Atomic Absorption Spectroscopy especially in the Part Per Billion (ppb) level.

The disadvantages of microwave digestion is associate with it limitation to sample amount, which play a critical role especially when analyzing trace metal in edible oil whereby high sample amount maybe preferable. Apart from that, Microwave digesters are usually costly and expensive, therefore other optional preparation methods are sometimes chosen in comparison to microwave digester.

3.2 VOLTAMMETRY AND POLAROGRAPHY

The term Voltammetry was first used by kolthoff in 1940, it refer to a group of technique that fall under electrochemistry, which utilized the potential or voltage in the unit of Volt that applied to an electrode system, and measure the current in the unit of ampere that flow through the chemical cell. Polarography was the term that use, when the voltammetry technique is applies to a working electrode system whereby a mercury is flow through a capillary to form a droplet (dropping mercury electrode), and a potential current relationship is measured. The technique of Polarography was introduce by Heyrovsky and the word polarography was come from the name polarized, which mean that no current are able to flow across the interface (Mercury) of the working electrode with solution although there is a potential different across the electrode. In order to prevent confusion, IUPAC define Voltammetry as a general term that used when current potential relationship is measured, while Polarography term is used when the flowing

conducting liquid electrode such as dropping mercury electrode is used as the working electrode for investigating the current potential relationship.

3.2.1 PRINCIPLE AND INSTRUMENTATION OF VOLTAMMETRY

In the early development of voltammetry and polarography instrument, the system is basically consist of simple setup that comprise a direct current Potential Source (potentiostat), an Ammeter for current measurement and a measuring cell. Two type of electrode system was used in the early cell which is know as the counter electrode and the working electrode, both electrode system was immersed in the ionic solution that contain the substances of analytical interest and electrolytic conductance. This ionic solution will complete the electric circuit of the whole set up, therefore when a voltage range is applied to the working electrode, the resulting current is able to measure by the counter electrode. The information and magnitude of the current response provides the evidence for which quantitative and qualitative analysis can be perform.

The simple 2 electrode set up of the system does not provided a good outcome, and as well limit the application of the system. In Basic, the counter electrode had 2 purposes, the first function is to act as a reference electrode and the second function is to complete the circuit by carrying the current that generate at the working electrode during the chemical process. The problem arise was due to the counter electrode used, which cannot provide a stable and precise control of working electrode potential in relative to the reference electrode being used. In order for the counter electrode to be used as an

ideal reference system, the counter electrode must be able to provide a constant potential and as well must be depolarizable but that is not the cases in the counter electrode used.

Therefore, in modern voltammetry system 3 electrode system is employed the counter electrode no longer function as both reference electrode and also as carrier for current that generated in the working electrode, instead a third electrode know as the auxiliary electrode (AE) is introduce which function to complete the current carrying circuit, the type of auxiliary electrode used are of platinum electrode. The figure 3.2 illustrated a simple set up of a 3 electrode system of a voltammetry instrument.

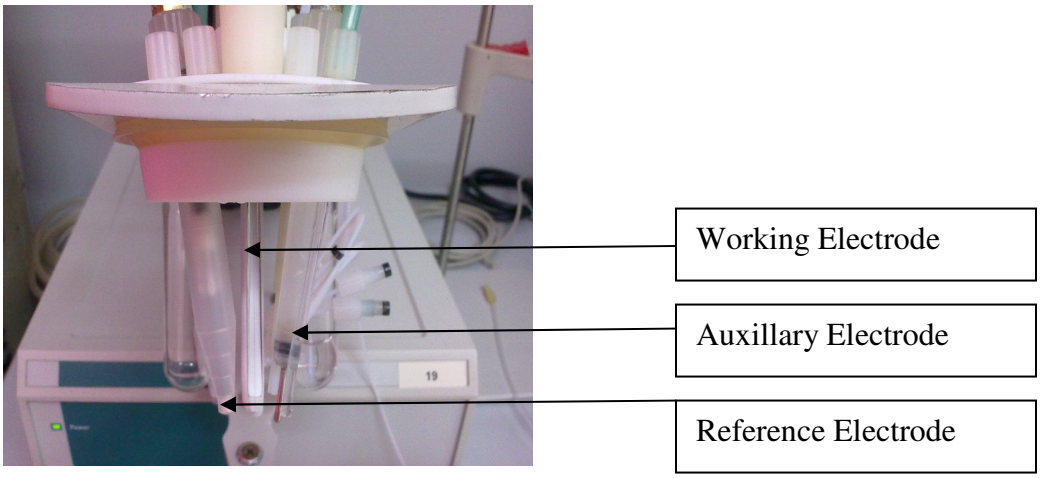


Figure 3.2 Voltammetry Instrument with 3 Electrode Systems

The introductions of the Auxiliary electrode mean that now the counter electrode only serve as one function as a true reference electrode. The construction of reference electrode can be of single or double junction and very depend on the type of sample is

measuring. Two of the well know references electrode that used in electrochemistry are the silver-silver chloride (Ag-AgCl) electrode in which the electrolyte used are the potassium chloride, the second type of is know as calomel electrode using a mercury electrolyte. The latter system is less preferable due to it hazardous of mercury content. The reference electrode system used provided a constant potential in order to ensure a precise voltage or potential is apply to the working electrode, the chemical reaction for a Ag-AgCl reference electrode is show as below:

$$AgCl \text{ ----------} \rightarrow Ag^{+} (solid) + Cl^{-} (liquid) \quad \text{Std potential} = 0.222v$$

The standard potential is only truth when 1 mol/L of potassium chloride (KCl) is used as the electrolyte. However at different concentration the potential can be calculated from Nerst equation:

$$\text{New Ref potential (v)} = \text{Std Potential} - RT/nf \text{ Log } [Cl^{-}]$$

Whereby, R= gas constant,

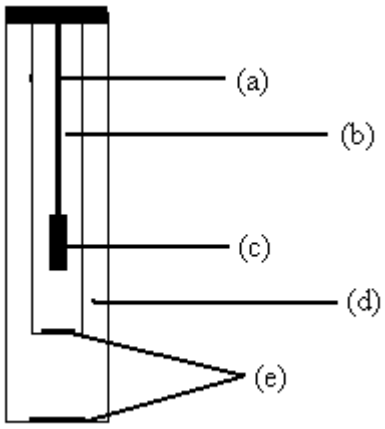
T = Temperature

n = Charge

f = Faraday constant

$[Cl^{-}]$ = concentration of Chloride in the electrolyte used

Two type of junction are usually available, a single junction (salt bridge) or double junction (salt bridge), a double junction system had an advantage of allowing two different electrolyte to be used, for instance if the chloride ion in the KCl causes interference to the measuring cell a second kind of electrolyte can be used on the outer compartment for example potassium nitrate (KNO3). The Figure (3.3) below illustrated an example of a double junction reference electrode system:



(a) = Silver Wire

(b) = KCl Electrolyte

(c) =Silver Chloride

(d) = second electrolyte (KCl, KNO3)

(e) = Salt bridge (junction)

Figure 3.3. Double Junction Silver-Silver Chloride Reference Electrode

The working electrode are the most important in the voltammetry this because various type of working electrode are available. In the cases mercury electrode is used as a working electrode the term polarography is used to refer the system. In general the working electrode can be divided into two types the liquid or solid working electrode. The only type of liquid working electrode is the mercury electrode, whereas for solid electrode there is various type example are rotating platinum disk electrode (RDE), Glassy carbon electrode (GCE), carbon paste electrode (CPE), gold electrode and silver

electrode. The solid working electrodes are often used for cyclic voltammetry (CV) analysis.

Mercury working electrode can be divided into 3 types and is depend on the construction of the electrode itself. The 3 types are included, dropping mercury electrode (DME), hanging mercury dropping electrode (HMDE), static mercury dropping electrode (SMDE). The only different in this mode are the method of manipulating the mercury dropping. Each of the modes used account for it different sensitivity. In the case DME mode is used, often it is used for analysis in ppm range whereas for SMDE is for sub ppb range and for HMDE is for ppb and Part per Trillion (ppt) analyses. In order for the analysis using HMDE the voltammetry technique used often is by stripping voltammetry technique. The sensitivity of DME is in ppm range due to the dropping mercury is inconsistency as a result of the surface area of the mercury drop produce is continuously changing and not constant. As a result a more improvised mode knows as SMDE was introduced which produce a more constant surface area of the dropping mercury. The construction of a SMDE working electrode is incorporated a valve system which can open and shut at a short time of 20-200 milisecond, therefore this produce a mercury drop which is more constant in surface area. In the case for HMDE mode the working electrode form a small drop of mercury which hang on the tip of the capillary, this mode produce a very constant surface area and long hanging time that allow stripping voltammetry technique to be used perfectly. The figure 3.4 shows the hanging dropping mercury electrode.

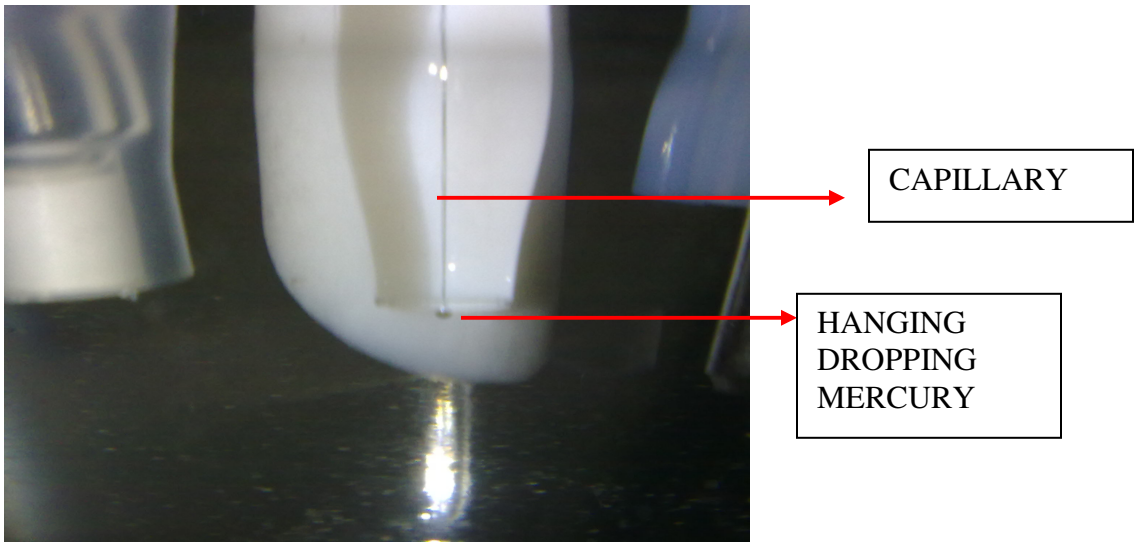


Figure 3.4 Hanging Dropping Mercury (HMDE)

Today modern mercury working electrode is design to be able to switch between the 3 different modes of analysis. However the disposable of mercury waste are sometimes a problem to the usage of voltammetry analysis, as a solution to the problem above some manufacture had introduced the used of a thin film mercury electrode, in which mercury is electrolytically deposited onto a solid electrode such as glassy carbon or iridium prior to analysis, this type of electrode is suitable for stripping voltammetry and chronopotentiometric analysis. However dropping mercury electrode is preferred in some instance that a new surface is form in each mercury drop which makes it an advantage against the thin film mercury electrode. The figure 3.5 illustrated a schematic diagram of mercury dropping working electrode.

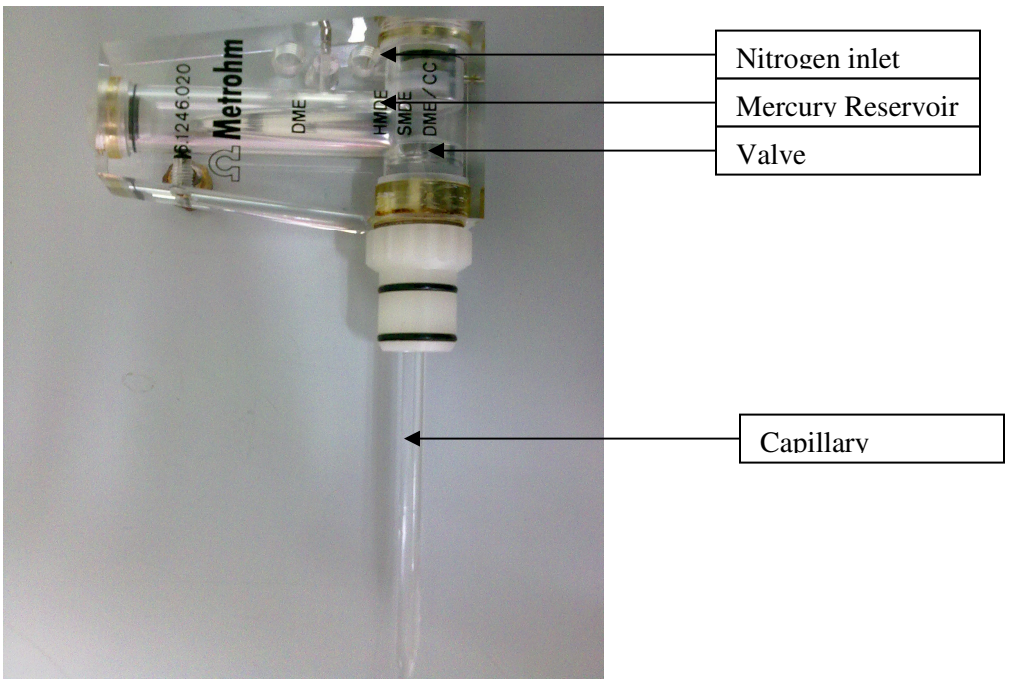


Figure 3.5 Multi-Mode Mercury Working Electrode

The most classical technique of polarography was know as direct current polarography (DCP) which the current that flow through the working electrode is measure in corresponding to a linear voltage alteration, in which the chemical reaction (oxidation or Reduction) is occur at the working electrode. On closer observation the current that flow through the working electrode can be divided into faradaic current (I_f) and capacitive current (I_c). The Capacitive current is basically occur due to the charging and discharging or the electrochemical double layer on the surface of the working electrode. In most polarographic analysis the Faradaic current is the measuring signal that we want, and the capacitive current is the unwanted component due to it interferences to the faradaic current, therefore the ratio of (I_f) to (I_c) is important as it give a sensitivity measure of the

polarographic. Many technique had been introduced in order to maximize the ratio so that the faradaic current is maximize while capacitive current is reduce as low as possible, some of the technique that been developed in polarography included sampled Direct Current Polarography (DCP) and Pulse method which emphasized on reducing the capacitive current, and Stripping Voltammetry method which attempt to increase the faradaic current in order to increase the (I_f) to (I_c) ratio. All this technique used apply to different mode of working electrode this is DME, SMDE, and HMDE. In this dissertation, only the principle of Stripping Voltammetry is highlighted due to this technique is used for the analysis of nickel, cadmium, lead and zinc.

3.2.2 STRIPPING VOLTAMMETRY

Stripping voltammetry techniques are the most efficient electrochemical technique for trace metal analysis. In comparison to the conventional direct current polarography, the stripping voltammetry technique is highly sensitive by a factor of 10^3 to 10^5. This technique can have a detection limit of between 10^{-9} to 10^{-11} mol/L.

Stripping Voltammetry is a technique in polarography which utilized the hanging mercury dropping electrode (HMDE) mode for analysis. The principle of stripping voltammetry is basically can be divided into two steps. The first step is know as deposition step in which a constant deposition potential is apply to the working electrode and can be controlled by a time factor, as a result of the constant potential the analyte to be analyze will be electrolytically deposited to the Hanging mercury working electrode

either as amalgam, or sparing soluble mercury compound or adsorptive as a complex compound. On the second step is know as stripping step in which a potential sweep is apply either cathodically or anodically which resulting in the reduction or oxidation process at the working electrode, as a result of this the analyte will strip out from the working electrode as anion or cation into the electrolyte and a diffusion current is produce which flow through the working electrode. There is 3 common type of Stripping Voltammetry technique namely Anodic Stripping Voltammetry (ASV), Cathodic Stripping Voltammetry (CSV) and Adsorptive Stripping Method (AdSV). In some cases often term such as DPASV or SWASV is used which it indicated the mode of potential is used. The potential mode that apply in the stripping step are usually of differential pulse (DP) or square wave (SW), instead of linear potential sweep

The main different among the 3 technique is in term of it deposition and stripping characteristic. In Anodic Stripping Voltammetry, the metal or element that analyze must be able to form amalgam with the mercury at the working electrode for example metal such as Zinc, Cadmium, Lead, Chromium, Copper can be analyze with the ASV technique. In the first step, during deposition potential often a very negatively potential is applied for example such as at -1.5v. At this potential the metal of interest will undergo cathodic reduction to form amalgam with the mercury (Me^0Hg). At this step, the deposition will occurs under control stirring and at an optimum deposition time in order to ensure the metal is deposited to the working electrode. The deposition time play an important role for the optimization of the analysis for example at a low concentration of metal (ppb) the deposition time had to be increase to ensures sufficient amount of metal

is deposited to the working electrode. In the second step, a potential sweep (differential pulse) is applied to the working electrode at a more positively potential direction such as from -1.2v to -0.1 V. At this step, the metal will undergo oxidization (anodic) from the amalgam to form free ion again (Me^{n+}). At the stripping step, often the process takes place at a non stirring condition. The Mechanism of Anodic Stripping Voltammetry (ASV) can be summarized as below:

$$Me^{n+} + Hg + ne^{-} \underset{\text{Stripping (anodic)}}{\overset{\text{Deposition (cathodic)}}{\rightleftharpoons}} Amalgam(Me^{0}Hg)$$

In the analysis of Cadmium, Zinc and Lead in the edible oil sample, the anodic stripping voltammetry technique will be used. Lead and Cadmium will be analyzed in one voltammogram scan, however due to Zinc often give a very sharp and large peak, the zinc is analyse separately from cadmium and lead. Lead will strip at a potential of – 0.40v and cadmium at – 0.60v therefore a deposition potential of -0.800v will be apply to the working electrode, the deposition time will be varied depend on the concentration of the metal in the sample. For the cases of Zinc, a separate scan will be perform and by using the same sample solution, as the Voltammetry is a non destructive technique. Zinc stripped at a potential of approximate -1.00v and therefore the deposition potential is set at -1.20v. For both cases, the acetate buffer will be used as the electrolyte or supporting solution. The function of the acetate buffer is important to adjust the pH of the solution in order for the particular electrode reaction of the metal to occur. The diffrential pulse (DP) is choose as the voltage modulation mode during the stripping or determination step.

In Cathodic Stripping Voltammetry (CSV), the technique only can be applied provided that the analyte or metal in the sample can form a sparing soluble metal with mercury salt. The cathodic stripping voltammetry is very similar to anodic stripping voltammetry, in such a way that the process is the reverse mechanism of anodic stripping voltammetry. It also consists of two step, during the deposition step the analyte or metal which usually an inorganic or sometimes can be a organic anion will be anodically deposited to the mercury working electrode as a sparing soluble mercury (I) salt (Hg_2M_n). In order to deposited, the mercury of the working electrode need to be oxidized usually a more slighty negative potential is apply, in most of the case the deposition potential is set at the range of -0.2v to +0.4v (against Ag-AgCl / 3 M KCl Reference electrode). The deposition potential setting is very much depend on the electrolyte used this is because at different electrolyte the mercury oxidized at a different potential. On the second step which is the stripping step a cathodic potential is apply to reduce the mercury (I) salt (Hg_2^{2+}) back to mercury metal (Hg^0). Halides, Pseudohalides, Oxometallates, and organic anions can be analyzed by this methods, however as in each cases the determination process is based on the reduction of the mercury ion (Hg_2^{2+}) therefore only a same potential peak will be observed. The mechanism of Cathodic Stripping Voltammetry (CSV) can be summarized as follow:

Deposition step:

Anodic (Oxidation)

$$2\,Hg \leftarrow\text{----------}\rightarrow Hg_2^{2+}$$

$$Hg_2^{2+} + nM^- \leftarrow\text{----------}\rightarrow Hg_2M_n$$

Determination Step (Stripping)

Cathodic (Reduction)

Hg_2M_n ←----------→ $2Hg + nM^-$

One of the applications of Cathodic Stripping Voltammetry is the analysis of Arsenic; other application to organic anion included as well Mercaptan, Thiols, cysteine, Thiourea, Thioamine and many more. In Some cases Cathodic Stripping Voltammetry analysis can be used to determine several elements by adding some solution partner into the sample solution which function as an intermetallic compound, one of the applications is the analysis of arsenic, tellurium and selenium by using Copper (II) as the intermetallic compound.

In Adsorptive Stripping Voltammetry, the deposition of this method is indirect and does not involve the formation of amalgam; in fact the accumulation is done by adsorptive to the mercury of the working electrode. This method is an added advantages to stripping voltammetry due to the fact that many metal such as nickel, Iron, Aluminium, Cobalt, Titanium, uranium, platinum, tungsten, molybdenum, and many more does not form amalgam easily with mercury and also due to that many of the reaction is irreversible. Adsorptive striping voltammetry utilized a complexing agent or ligand which can form complex with the analyte of interest; the complexes are usually accumulated to the mercury by adsorptive process instead of a charge transfer process. As similar to ASV and CSV, the first step of Adsorptive Stripping Voltammetry involve the deposition of the complexing agent, but however the deposition step can be different in

mechanism and it is depend on the properties of the complexing agent used. One of the simplest mechanisms is the formation of complexes between the analyte of interest and the complexing agent, which only then follow by adsorption when a cathodic potential is apply, the mechanism is summarized as below:

Complex formation

M^{n+} + nL (complexing agent) ------→ ML^{n+} (dissolved)

Adsorption

ML^{n+} (dissolved) ----→ ML^{n+} (adsorbed)

Second deposition mechanism is based on adsorption of the complexing agent to the mercury, which is then followed by complexation with the analyte of interest. The mechanism can be summarized as below:

Adsorption

nL (complexing agent) ------→ nL (adsorbed)

Complex formation

M^{n+} + nL (adsorbed) ------→ ML^{n+} (adsorbed)

The magnitude of deposition potential is very depend on the physical properties of the complexing agent or ligand used. In general a neutral molecules (complexing agent) tend to adsorb to mercury at zero potential, this where the electrode charge is small and the electric field in the double layer (Helmholtz layer) is weak, in the case of anion molecule

which included those that have aromatic ring are generally adsorbed in the region of positive potential, and lastly the cation molecule which adsorbed in the negative region. Therefore, the accumulation potential is very much depend on the type of complexing agent used. The second step of the AdSV is the stripping of the complexing agent, which give a characteristic current peak in the current-voltage plot. The stripping potential apply is as well depend on the deposition potential of the complex-analyte adsorption.

The table 3.6 listed some of the common complexing agent or ligand that often used in Adsorptive Stripping Voltammetry.

Complexing agent or Ligand	**Application (Element)**
1,2 Dihydroxybenzene	Uranium, Copper, Iron, Vanadium, Germanium, Tin, Arsenic, Antimony
2,3-Butandione Dioxime (DMG)	Cobalt, Nickel, Palladium
8-Hydroxyquinoline	Copper, Cadmium, Lead, Uranium
N-Nitroso-N-Phenylhydroxylamine	Uranium, Molybdenum, Thallium
0-Cresol phthalexone	Cerium, Lanthanum, Praseodymium
Solochrome Violet RS	Aluminium, Iron, Galium, Magnesium, alkali and alkaline earth metal

Table 3.6 List of complexing agent or Ligand used in Adsorptive Stripping Voltammetry (AdSV)

One of the advantage of adsorptive Stripping voltammetry (AdSV) technique is in term of it detection limit, the AdSV can reach a determination limit in the ultra trace range to approximate 10^{-10} Mol/L, beside that the AdSV technique can be used as well for determination of surface active organic molecule with electro chemically active functional group.

The Analysis of nickel in Edible oil sample is performed by using the Adsorptive Stripping Voltammetry, the determination is done by using 2-3 Butanedione dioxime or also know as dimethylglyoxime as the complexing agent. The analysis is first initiate by formation of complexes (DMG-NI) in an alkaline pH condition usually ammonia buffer is used. A deposition potential of -0.7 is apply and under stirring condition the complex DMG-Ni is adsorbed to the hanging mercury surface. The determination step is performed by applying a differential pulse voltage sweep to cathodic range, this is from -0.7v to -1.15v, and the DMG-Ni will be desorbed from the surface and a peak current at potential -1.0 V will be observed. The concentration of Ni in the sample can be determined by using the standard addition technique. The figure 3.7 shows the structure of Bis-(dimethylglyoxime) Nickel (II) complexes.

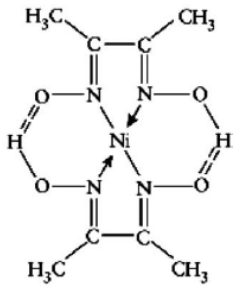


Figure 3.7 Chemical structure of Bis-(Dimethylglyoxime) Nickel (II)

4.0 REVIEW OF LITERATURE

In General various analytical and sample preparation methods had been propose for the analysis of metal in edible oil. Most of the analytical methods used are multi-metal analyses that can perform in a single run. The most frequent used technique is by Inductively Couple Plasma (ICP) either of Mass Spectrometry (MS) or Atomic Emission Spectroscopy (AES), other method also had been reported included Graphite furnace Atomic Absorption Spectrometric (GFAAS), and as well the Atomic Absorption Spectroscopy (AAS).

Although many analytical instrument techniques can be used in the analysis, however sample preparation is still an important procedure since the sample are edible oil which usually content high amount of carbon or organic. The amount of carbon content in edible oil are in the range of 70 to 80 % (Pier Luigi, Loretta, and jawahar, 2002) which required a proper and complete sample digestion or dissolution in order to de-mineralised or to obtain a free ionic species. Many preparation techniques had been used for the sample preparation in edible oil, the most commonly used are by microwave digestion, other technique included extraction by reflux especially for edible oil such as margarine, UV photolysis digestion, solvent extraction, dialysis and emulsified extraction. The table on next page listed some example of carbon content in various food types.

No	Food	Carbon Content
1	Vegetable Oil	70-80%
2	Vegetable Fats	70-80%
3	Wheat	45%
4	Starches	40-50%
5	Fruit	40%
6	Fish	52%
7	Egg	50%
8	Sugar	42%

Table 4.1 Carbon content (%) in various food types

From the table, vegetable oil and fats contain the highest amount of carbon in compare to other food, this is important because the information is vital especially in sample preparation, especially in determine the required amount of sample, reagent and the experiment condition.

Several methods had been developed for the analysis of metal in edible oil, one of the works was developed by Y.Sahan, Basoglu and Gucer whereby the research interest was only focus on olive oil that using microwave digestion and Inductively Couple Plasma Mass spectrometry. The olive oil sample was prepared with microwave digester and heated in the followed program (table 4.2) with 0.5g sample, 5ml of 65% HNO3 and 1 ml of 30% Hydrogen peroxide microwave digester.

Power (watt)	Ramp (min)	Hold	Fan
250	0.00	01.00	1
400	05.00	10.00	1
600	05.00	30.00	2
0		20.00	2

Table 4.2 microwave digestion heating program for Edible Oil

The digested olive oil sample was analyzed with Inductively Couple Plasma Mass Spectrometry (ICP-MS) method with radio frequency setting at 10MHz and sample flow at 1.8ml/min. The average result obtained for the following metal Zinc, Cadmium, Nickel and Lead in olive oil particular green olive oil and Black Olive oil was summarized in the table below:

METAL	BLACK OLIVE OIL Mg/Kg	GREEN OLIVE OIL mg/Kg
Zinc (Zn)	8.50 +/- 1.74	10.58 +/- 2.01
Cadmium (Cd)	0.11 +/- 0.01	0.12 +/- 0.04
Nickel (Ni)	0.30 +/- 0.06	0.37 +/- 0.06
Lead (Pb)	0.71 +/- 0.07	0.75 +/- 0.12

Table 4.3 Determination of Zinc, Cadmium, Nickel and Lead in Olive Oil with ICP-MS

From this work (Y.Sahan, Basoglu and Gucer 2007), Zinc are found to be highest concentration in the range of 8.00 mg/Kg to 11.00 mg/Kg, this is because zinc is a source of micronutrient which required by most plant in comparison to other metal (Cd, Ni and

Pb) which are considered as toxic. The concentration of the Cadmium, Nickel and Lead was reported at concentration of less than 1ppm.

A similar work was described by I.J Cindric, Michael Zeiner and steffan, whereby Graphite Furnace Atomic Absorption Spectroscopy (GFAAS) and Inductively Couple Plasma Atomic Emission Spectroscopy (ICP-AES) was used to analyze Aluminium (Al), Cobalt (Co), Copper (Cu) Nickel (Ni), Lead (Pb), Zinc (Zn), Iron (Fe), and Magnesium (Mg) in range of edible oil sample included pumpkin oil, olive oil, sunflower oil, soya oil and rice oil. The sample (0.5g) was prepared by microwave digestion as well. Table 2.6 below summarized the analysis work of Lead, Nickel and Zinc in the edible oil by I.J Cindric and Etc, 2007. The results reported are based on average result of 3 to 5 oil for each sample.

Sample	**Nickel mg/Kg**	**Zinc Mg/Kg**	**Lead Mg/Kg**
Olive Oil	1.60	3.40	<0.001
Pumpkin Seed	6.10	13.50	<0.001
Sunflower Oil	0.10	3.20	<0.001
Soyabean Oil	0.23	4.30	<0.001
Rice Oil	0.08	2.90	<0.001

Table 4.4 Determination of Nickel, Zinc and Lead in Edible Oil by GFAAS and ICP-AES

The analysis result describe by I.J cindric and etc, show that the concentration of zinc are the highest in comparison to other metal in the range of 3.0 mg/Kg to 13.0 mg/Kg which is in adjacent to that of report by Y.Sahan and etc. The nickels are found to

be the highest concentration in the Pumpkin seed oil and Olive oil, whereas compare to other sample are in the range of below 1ppm. The Lead is detected at concentration of less than 0.001 mg/Kg.

Inductively Couple Plasma (ICP) technique had an advantaged of multiple metal analysis and as well better detection limit. However Atomic Absorption (AAS) method is sometimes preferable by laboratory due to it low operation cost. In contrast to ICP method, analysis of metal in edible oil by AAS was worked by D.Mendal, M.Tuzen, M.Soylak, and Uluozlu. The Edible Oil sample was prepared by microwave digestion with 65% nitric acid and Hydrogen Peroxide. The following metal Copper, Zinc, Iron, and cobalt was analyze with AAS, while Cadmium and lead was analyzer with GFAAS. Table 4.5 showed the analysis result of the metal in edible oil namely Olive Oil, Sunflower oil, Hazelnut Oil, and Margarine Oil

Sample	Zinc (mg/Kg)	Cadmium (ug/kg)	Lead (mg/Kg)
Olive Oil	1.03 +/- 0.1	0.15 +/- 0.02	0.03 +/- 0.003
Hazelnut Oil	1.15 +/- 0.1	4.57 +/- 0.4	0.01 +/- 0.01
Sunflower Oil	1.10 +/- 0.1	3.76 +/- 0.4	0.01 +/- 0.01
Margarine	2.71 +/- 0.2	3.66 +/- 0.3	0.01 +/- 0.01

Table 4.5 Determination of Zinc, Cadmium and Lead in Edible Oil by Atomic Absorption Spectrometry (AAS)

The Atomic Absorption Spectrometry analysis in edible oil by D.mendel and etc illustrate that the cadmium and lead are found to be present at part per billion (ppb) levels only, which is expected to be low since both metal are known to be toxic. Although the

cadmium and lead was analyze with GFAAS which is only single element analysis, however it show a comparable detection limit to Inductively Couple Plasma (ICP). On other hand, Zinc is detected in the range of 1.0 mg/Kg to 3.0 mg/Kg. In another work by Mariela N. Matos Reyes and Reinaldo C.Campos, (2006) an analysis of nickel and copper by GFAAS was perform on edible oil mainly focus on Soybean Oil and Corn Oil, the average result was summarized as below in table 4.6.

Sample	Nickel mg/Kg	Copper mg/Kg
Soybean Oil	3.62	3.16
Corn Oil	4.19	3.07

Table 4.6 Determination of nickel and copper in edible oil by GFAAS

The nickel concentration reported was higher in comparison to the result reported by other literature (result table 4.3 and 4.4). This is reported at the range of 3.5 mg/Kg to 4.19 mg/Kg. The work described by Mariela N.Matis Reyes and et al only limited to nickel, as the sample are direct analyze by Graphite Furnace Atomic Absorption Spectroscopy (GFAAS) without sample pretreatment. Although the GFAAS technique is direct and can prevent loses due to sample pretreatment such as digestion, nevertheless the GFAAS method are not optimize for analysis of wide range of metal in edible oil analysis due to the problem of non-specific absorption as a consequence of edible oil sample matrixes (I.Karadjova, G.Zachariadis, G. Boskuo and J.Stratis, 1998).

The overall analysis of metal by ICP and AAS technique show a regularity result when microwave digestion technique was employed as the sample preparation method.

However, similar work was also reported but with different sample preparation technique particularly by extraction method instead of microwave digestion. Although microwave technique is fast and reliable, nevertheless the microwave digesters are costly and maybe not affordable by some laboratory, in contrast to extraction technique which is more easily set up and at lower cost.

One of the works that employ the extraction procedure in edible oil sample pretreatment for metal analysis was describe by Erol Pehlivan, Gulsin Arslan, F.Gode, and et al (2008), whereby the edible oil sample was extracted with 10% dilute nitric acid shaken at water bath (50 C) for 2 hours, then follow by centrifugation at 2800 rpm. The acid aqueous layer was then removed and dilute in 25ml volumetric flask. The prepared sample was analyzed with Inductively Couple Plasma Atomic Emission Spectroscopy (ICP-AES) for the following metal Copper, Iron, Lead, Zinc, Cadmium, Chromium, Cobalt, Manganese and Nickel. Table 4.7 summarized the average analysis result of the metal in various type of edible oil sample.

Sample	**Zinc mg/Kg**	**Cadmium Mg/Kg**	**Lead mg/Kg**	**Nickel mg/Kg**
Olive Oil	0.0512	0.0021	0.0017	0.0018
Sunflower Oil	0.0319	0.0017	0.0013	0.0032
Corn Oil	0.0294	0.0009	0.0000	0.0062
Almond Oil	0.2870	0.0003	0.0000	0.0254
Hazelnut Oil	0.0185	0.0012	0.0000	0.0072
Soybean Oil	0.0348	0.0013	0.0000	0.0027

Table 4.7 Determination of Zinc, Cadmium, Lead and Nickel in Edible Oil by sample extraction and with ICP-AES

From the result, sample that prepare by extraction method showed a considerable lower concentration of zinc and nickel in the edible oil, in comparison to sample that prepare by microwave digestion. The overall concentration of the Zinc, Cadmium, Lead and Nickel is less than 100 part per billion (ppb) except for Zinc in almond oil sample which is about 0.2870 mg/Kg.

In a similar case, described by Giacomo Dugo, Lara la pera, G.L La Torre and D. Giuffrida (2004) the same extraction approach is used to prepared the edible oil sample, electrochemistry technique know as Derivative potentiometric Striping analysis (DPSA) is employ. The extraction procedure is perform with 2ml of 35% hydrogen peroxide and 10ml of 36% hydrochloric acid. The extraction process is perform by using magnetic stirrer for about 30 minute at temperature of 90 degree Celsius, then follow by centrifugation the aqueous layer is diluted at volumetric flask, and the organic layer is further extract. The prepared edible oil sample was analyzes using DPSA technique for Cadmium, Copper, Zinc and lead. The table 4.8 summarized the average concentration of the metal in edible oil by DPSA method. The result describe by Giacomo Dugo and Etc show that the concentration of the zinc, cadmium and lead are in the range of less than 100 ppb except for some edible oil sample which show a slightly higher concentration of Zinc.

Sample	Zinc (ug/Kg)	Cadmium ug/Kg)	Lead ug/Kg)
Sunflower Oil	232.76	1.72	9.98
Maize Oil	51.54	4.92	8.32
Peanuts Oil	396.1	2.69	10.00
Rice Oil	192.4	1.27	8.60
Soy Oil	37.76	3.75	25.48

Table 4.8 Determination of Zinc, Cadmium and Lead in Edible Oil by Derivative Potentiometric Stripping Analysis (DPSA).

5.0 METHODOLOGY

The method used in this analysis can be divided into 2 parts which is the sample preparation part and sample analysis part. As described above, the sample contain a high organic matrixes which must be digested before sample analysis, a microwave digester will be used for the sample preparation. The digested sample is kept in sample bottle and labeled before analyze in the lab by voltammetry method.

5.1 APPARATUS AND INSTRUMENT

a) PRECISA Analytical Balance (0.001g)

b) CEM Microwave Digester, MARS XPRESS

The following accessories was used –

i) 8 Sample Vessel Carousel

ii) Polyflouroacetate (PFA) Sample Vessel

c) METROHM 797 VA (VOLTAMMETRY) COMPUTRACE

The system equipped with 3 electrodes –

i) Metrohm multimode Mercury Dropping Electrode for DME, SMDE and HMDE mode.

ii) Platinum Auxillary electrode

iii) Silver-Silver Chloride Reference Electrode

d) Milipore Ultrapure Water (UPW) System

e) Micropipette 10-100ul

f) Micropipette 100-1000ul

g) Volumetric Flask 10ml

h) Volumetric Flask 100ml

i) Volumetric Flask 250ml

j) Volumetric Flask 50ml

k) Beaker 100ml

l) Volumetric Pipette 10ml

m) Measuring Cylinder 100ml

n) Measuring Cylinder 10ml

o) Wash Bottle

p) Glass Funnel

q) Sample bottle 50ml with PTFE Cap

r) Whiteman 0.45um Filter Paper

5.2 CHEMICAL

The chemical that used for microwave digestion and voltammetry were listed as below:

a) Hydrogen Peroxide 30%, H_2O_2

b) Concentrated Nitric Acid 70%, HNO3

c) Ultrapure Water (18MOhm)

d) Potassium Chloride, KCl

e) Glacial Acetic Acid 99.0%, CH_3COOH

f) Sodium Hydroxide 30%, NaOH

- 30 g of NaOH pellet is weighed with analytical balance and transfer into a 100ml beaker. By using a measuring cyclinder, 100ml of ultrapure water was measured and transfer into the beaker. The Solution was stirred until all the pellet was complete dissolved.

g) Ammonium 25%, NH_4

h) Hydrochloric Acid 37%, HCl

i) DimethylGlyoxime

j) Ethanol 95%, CH_3CH_2OH

k) Lead 1000ppm Standard, Pb (Metrohm)

l) Zinc 1000ppm Standard, Pb (Merck)

m) Nickel 1000ppm Standard, Pb (Merck)

n) Cadmium 1000ppm Standard, Cd (Merck)

o) 3M Potassium Chloride, KCL Electrolyte For Reference Electrode (METROHM)

p) Mercury (Fluke), for Working Electrode

q) Nitrogen Gas 99.999% purity, N_2

5.2.1 PREPARATION FOR POTASSIUM CHLORIDE ACETATE BUFFER (KCl-ACT)

1) 28.00g of Potassium Chloride powder was measure with Analytical Balance and transferred into a beaker. By using measuring cylinder 100ml of ultrapure water was measured and transfer into the beaker. The solution was stirred until the potassium chloride was completely dissolved.

2) The Dissolved potassium chloride solution was transferred into a 250ml volumetric flask.

3) 12.50ml of 30% Sodium Hydroxide and 7.1ml of Glacial Acetic Acid was measured and transferred into the 250ml volumetric flask. The solution was top up to 250ml with ultrapure water and shake to mix the solution.

5.2.2 PREPARATION OF AMMONIA CHLORIDE (NH_4Cl) BUFFER

1) A 250ml Volumetric Flask was quarterly filled with ultrapure water. 11.04ml of Concentrated Hydrochloric Acid was carefully measured and transfer into the volumetric flask.

2) By using a measuring cylinder 56.25ml of Ammonia 25% was measured and transferred carefully into the 500ml volumetric flask under fume cupboard.

3) The Solution was top up to 250ml with ultrapure water, and shake well to mix the solution.

5.2.3 PREPARATION OF DIMETHLYGLOYXIME (DMG) IN ETHANOL

1) 1.17g of DMG was weighed using analytical balance and dissolved in Ethanol. The solution was stirred slowly until all the powder is dissolved.

2) The solution was then fill up to 100ml with ultrapure water

5.3 SAMPLING AND SAMPLE PREPARATION OF EDIBLE OIL SAMPLE

As a common analysis procedure, prior to sample digestion the sample was first labeled in details for each sampling bottle, as well the carousel position and the PFA Sample vessel. Most of the samples used are from commercial ready to used product that available in Malaysia.

5.3.1 SAMPLING OF EDIBLE OIL

Two type of edible oil product was selected for these analyses, which are the Cooking Oil and the Margarine sample. Since for both product, various source of edible oil had been

used to produce the product, therefore only four type of edible oil source will be focused which are the Sunflower Oil, Palm Oil, Corn Oil and Olive Oil. For the cases of olive oil, margarine sample are only available as a mix product as a result of this, only the cooking oil will be selected for the analysis. The table 5.1 summarized the types and source of edible oil that had been purchase from commercial available product for the analysis.

The physical properties of Margarine and cooking oil are different in term of it morphological as well it melting temperature. For the cooking oil sample basically less sampling and preparation is required as the sample are present as a liquid form which the chemical composition are more homogenized and are in ready to used form. Whereas in the cases of margarine sample, the sample was present as a solid phase therefore sampling and sample preparation is an important step.

No	**Type of Products**	**Edible Oil Source**
1	Cooking Oil	Palm Oil
2	Cooking Oil	Extra Virgin Olive Oil
3	Cooking Oil	Corn Oil
4	Cooking Oil	Sunflower Oil
5	Margarine	Palm Oil
6	Margarine (Baking)	Palm Oil
7	Margarine	Corn Oil

Table 5.1 Type of product and Edible Oil source used for analysis

5.3.1.1 SAMPLING OF MARGARINE SAMPLE

1) By using a Spatula, the margarine sample was divided into 4 sections, in each section the sample was sampled from top, medium and based from the sample pack.

2) Each portion of the sample was transferred into a beaker. A water bath was prepared at temperature of approximately 60 degree Celsius, and the beaker contained sample was immersed to melt the solid sample into liquid form.

3) The sample was weighed and transferred into the PFA sample vessel (describe in section 5.3.2)

5.3.1.2. SAMPLING OF COOKING OIL SAMPLE

1) The oil sample was shake well in the bottle, by using a graduated pipette the sample is weighed accurately into the PFA sample vessel (Describe in Section 5.3.2)

5.3.2 SAMPLE DIGESTION BY MICROWAVE DIGESTER

1) 1.5-2.0 g sample was accurately weight using an analytical balance into the PFA Sample vessel, by using a clean graduated pipette.

2) 5ml of concentrated Nitric Acid 70% was measured with a volumetric pipette and transfer into the sample vessel carefully.

3) 2ml of hydrogen peroxide 30% was measure with a volumetric pipette and transfer into the sample vessel carefully.

4) The PFA sample vessel was closed with a plug and a vent cover, and the vessel was tighten with hand. The vessel was then placed into the sleeved before arranged it in the sample carousel.

5) A blank sample (method blank) was prepared as well following step 1 -4 without any sample added only contain Nitric acid and Hydrogen Peroxide.

6) All the PFA sample vessel is labeled, the sample weigh and sample information (sample name) was recorded.

7) The carousel was installed into the microwave digester chamber; the programmed which had been set early was recall and loaded into the system, and the digestion process is start. The table 5.2 below summarized the Microwave digestion time programming.

Microwave digester Parameter	Setting
Power (Watt)	800watt
Percentage power (%)	75%
Ramp Time (min)	15.00 min
Hold time (min)	10.00 min
Final temperature	200 degree Celsius

Table 5.2 Microwave Digester parameter setting for edible oil sample digestion

8) Once the digestion was completed, the sample vessel cap was release slowly to allow the acid fume vapor pressure to escaped from the vessel, the sample was then filtered through a 0.45um filter paper and transfer into a 25ml Volumetric Flask via a glass funnel.

9) The volumetric flask was top up to 25ml with ultrapure water, and shake well before transferred into labeled sample bottle.

5.4 PREPARATION OF CALIBRATION STANDARD

Freshly prepared calibration standard was used for through out the analysis procedure. The Cadmium and Lead standard was prepared as a mix standard, whereas for the Zinc and Nickel the standard was prepared individually from the cadmium and lead.

5.4.1 PREPARATION OF 1.00 PPM MIXES CADMIUM AND LEAD STANDARD

1) A 100ml volumetric flask was half filled with ultrapure water.

2) By using a Micropipette, 1000ul of cadmium and Lead stock standard (1.0g/L) was pipette and transfer into the 100ml volumetric flask.

3) 0.18ml of 30% nitric acid was pipette and transfer into the volumetric flask and top up to 100ml with ultrapure water. The solution standard shall contain 0.014mol/L of nitric acid.

5.4.2 PREPARATION OF 1.00 PPM ZINC STANDARDS

1) A 100ml volumetric flask was half filled with ultrapure water.

2) By using a Micropipette, 1000ul of Zinc stock standard (1.0 g/L) was pipette and transfer into the 100ml volumetric flask.

3) 0.18ml of 30% nitric acid was pipette and transfer into the volumetric flask and top up to 100ml with ultrapure water. The solution standard shall contain 0.014mol/L of nitric acid

5.4.3 PREPARATION OF 1.00 PPM NICKEL STANDARDS

4) A 100ml volumetric flask was half filled with ultrapure water.

5) By using a Micropipette, 1000ul of Nickel stock standard (1.0 g/L) was pipette and transfer into the 100ml volumetric flask.

6) 0.18ml of 30% nitric acid was pipette and transfer into the volumetric flask and top up to 100ml with ultrapure water. The solution standard shall contain 0.014mol/L of nitric acid

5.5 PREPARATION OF SPIKE SAMPLE WITH NICKEL, ZINC, LEAD AND CADMIUM STANDARD.

3 samples were spiked with the following metal Nickel, Zinc, Lead and Cadmium. Since the samples are present in a small quantity (25ml) therefore a 10ml volumetric flask was used.

1) A 10ppm Mix Standard of Zinc, Cadmium, Lead and Nickel was prepared by pipette 1.0ml of each 1000ppm standard into a 100ml Volumetric flask, and then top up to 100ml with ultrapure water

2) A 10ml Volumetric Flask was half filled with Sample (after digested sample) from step 5.3.2.

3) By using a micropipette, 100ul of 10ppm mix standard (prepared in step 1) was transferred into volumetric flask, and the solution was top up to 10ml with sample.

4) Step 1 to 3 was repeated for the other 2 sample.

5.6 PREPARATION OF VOLTAMMETRY INSTRUMENT

5.6.1 CALIBRATION OF VOLTAMMETRY INSTRUMENT WITH DUMMY CELL

As suggested by Metrohm procedure, the instrument system was checked with an electronic dummy cell to ensure the system is function as accordingly to the specification.

1) The connector cable of the 3 electrode system (working electrode, Auxiliary Electrode and Reference Electrode) was dismantle and connect to an electronic dummy cell as accordingly to the label.

2) From the Voltammetry software, the dummy cell method was loaded and the start button was click to execute the analysis

3) A Curve of Current against Voltage will be plot, and the result was recorded.

5.6.2 ELECTRODE CHECK

5.6.2.1 Reference Electrode

1) The outer filling electrolyte of the Ag/AgCl Reference electrode was removed from the electrode.
2) A fresh Potassium Chloride 3Mol/L Solution was filled into the outer compartment of the electrode.

5.6.2.2 Working Electrode

1) From the Voltammetry instrument software the manual control was click, the Hanging mercury dropping (HMDE) mode was selected, and a new drop button is click to allow mercury to drop and hang on the working electrode.

2) Step 1 was proceed few time, and the formation of hanging mercury was observed to ensure fresh mercury with constant size is form on the working electrode with every times the new drop button is click.

5.7 ANALYSIS OF ZINC, LEAD AND CADMIUM IN SAMPLE WITH ANODIC STRIPPING VOLTAMMETRY

1) The voltammetry glass vessel was cleaned with ultrapure water for few times.

2) 10ml ultrapure water was accurately pipette and transfer into the glass vessel.

3) 0.25ml of KCL-Acetate Buffer was accurately added into the glass vessel by using a micropipette.

4) 0.1ml of sample is accurately added into the sample vessel by using micropipette. The total Volume of the solution was 10.350ml.

5) The vessel is mounted into the voltammetry stand, and the electrode is lowered down into the sample until the lid was fully covered the vessel to ensure the vessel is air tight.

6) The Electrode was checked to ensure it is immersed in the sample, and no air bubble was formed in the solution.

7) From the voltammetry software the operating condition was set as following:

No	Parameter	Setting
1	Analysis Mode	Anodic Stripping Voltammetry
2	Mode	Differential Pulse
3	Working Electrode Mode	HMDE
4	Deposition Potential	- 1.1 V
5	Deposition time	30 s
6	Start Potential Sweep	- 0.9 V
7	End Potential Sweep	- 0.3 V
8	Sweep Rate	0.02 V/s
9	Pulse Amplitude	0.05 V
10	Pulse time	0.04 s
11	Purge Time	300 s
12	Stiring	2000 rpm

Table 5.3 Operation Condition for Cadmium and Lead analysis by Anodic Stripping Voltammetry.

8) Once the operation condition is saved, the analysis was started. The deposition time condition was subjected to change due to different analyte concentration.

9) Standard addition technique was performed with 0.100 ml of 1.00ppm mix Cadmium and lead standard was added into the vessel, upon requested by the voltammetry. 3 times standard addition was performed in each analysis.

10) Once the analysis is completed, the result was recorded.

11) The analysis was continuing with Zinc determination, the following operation condition was set as followed.

No	Parameter	Setting
1	Analysis Mode	Anodic Stripping Voltammetry
2	Mode	Differential Pulse
3	Working Electrode Mode	HMDE
4	Deposition Potential	- 1.5 V
5	Deposition time	10 s
6	Start Potential Sweep	- 1.5 V
7	End Potential Sweep	- 0.8 V
8	Sweep Rate	0.02 V/s
9	Pulse Amplitude	0.05 V
10	Pulse time	0.04 s
11	Purge Time	20 s
12	Stiring	1000 rpm

Table 5.4 Operation Condition for Zinc analysis by Anodic Strippping Voltammetry

12) Once the operation condition was saved, the analysis started.

13) Standard addition technique was performed; 0.100 ml of 1.00ppm Zinc standard was added into the vessel upon requested by the voltammetry. 3 times standard addition was performed in each analysis.

14) Once the analysis is completed, the result was recorded.

15) The sample was discharge into the waste bottle and the mercury droplet was dispose into the mercury waste. The glass vessel and electrode was clean with ultrapure water. The analysis was repeated for all samples, blank and spike sample.

5.8 ANALYSIS OF NICKEL IN SAMPLE WITH ADSORPTIVE STRIPPING VOLTAMMETRY

1) The voltammetry glass vessel was cleaned with ultrapure water for few times.

2) 10ml ultrapure water was accurately pipette and transfer into the glass vessel.

3) 1.0 ml of Ammonia Chloride Buffer was accurately added into the glass vessel by using a micropipette.

4) 0.1ml of sample is accurately added into the sample vessel by using micropipette.

5) 0.1ml of Dimethylglyoxime (DMG) was accurately pipette into the vessel by micropipette. The total Volume of the solution was 11.200ml.

6) The vessel is mounted into the voltammetry stand, and the electrode is lowered down into the sample until the lid was fully covered the vessel to ensure the vessel is air tight.

7) The Electrode was checked to ensure it is immersed in the sample, and no air bubble was formed in the solution.

8) From the voltammetry software the operating condition was set as following:

No	Parameter	Setting
1	Analysis Mode	Adsorptive Stripping Voltammetry
2	Mode	Differential Pulse
3	Working Electrode Mode	HMDE
4	Deposition Potential	- 0.1 V
5	Deposition time	5 s
6	Start Potential Sweep	- 0.8 V
7	End Potential Sweep	- 1.15 V
8	Sweep Rate	0.015 V/s
9	Pulse Amplitude	0.05 V
10	Pulse time	0.04 s
11	Purge Time	300 s
12	Stiring	2000 rpm

Table 5.5 Operation Condition for Nickel analysis by Adsorptive Stripping Voltammetry.

9) Once the operation condition is saved, the analysis was started. The deposition time condition was subjected to change due to different analyte concentration.

10) Standard addition technique was performed; 0.100 ml of 1.00ppm Nickel standard was added into the vessel upon requested by the voltammetry. 3 times standard addition was performed in each analysis.

11) Once the analysis is completed, the result was recorded.

12) The sample was discharge into the waste bottle and the mercury droplet was dispose into the mercury waste. The glass vessel and electrode was clean with ultrapure water. The analysis was repeated for all samples, blank and spike sample.

6.0 RESULTS AND DISCUSSION

An electronic Calibration analysis was performed on the voltammetry system prior to sample analysis. A Dummy Cell which is an electronic circuit box that function to stimulate the three electrode cable namely Working Electrode, Reference Electrode and Auxillary electrode cable, the Voltammogram below show the Dummy Cell (stimulation) result.

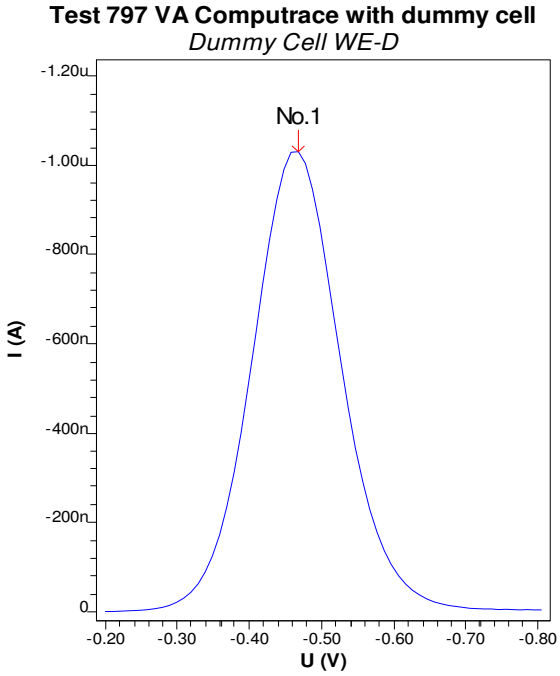


Figure 6.1 Voltammogram test result from 797VA computrace dummy cell

From the result, the peak current was 1.03 uA at voltage position of 0.47V, which the theoretical should be 1.00 with tolerance of 0.05 uA (5%) and position at 0.5 V with tolerance of +/- 0.05V. However the result was acceptable as it still within the tolerance range.

A total of 7 samples from different edible oil products (off shelf) was purchase from market and analyze. The sample that used was weighed and digested by using microwave digestion method. 3 samples were spike with standard for recovery study and a reagent blanks were prepared and analyze. For each of the analysis a minimum of two Scan was performed and the voltammogram average result is taking for report. The table below tabulated the details of blank result for Cadmium, Lead, Zinc and Nickel.

Blank	**Cadmium (ug/L)**	**Lead (ug/L)**	**Nickel (ug/L)**	**Zinc (ug/L)**
Blank 1	38.586	27.242	103.289	75.314
Blank 2	39.739	27.796	96.432	64.080
Average	39.163	27.519	99.861	69.697
Standard Deviation	0.815	0.392	4.849	7.944

Table 6.2 Concentration (ug/L) of Nickel, Cadmium, Lead and Zinc in Reagent Blank by Stripping Voltammetry

The concentration of the metal in the blank was found to be acceptable, this is because the chemical used was analytical (AR) grade although suprapure grade is recommended but due to unavailable therefore AR grade was used instead. The nickel in the blank was found to contain the highest with average concentration of about 99.861ug/L. whereas for other metal especially Cadmium and Lead are found at concentration range of below 40 ug/L. In the cases for Zinc, the average concentration was 69.697ug/L. The Limit of

Detection (LOD) was taken as 3 times the standard deviation of the blank, therefore the LOD for each metal are as below:

No	Metal	Limit of Detection (ug/L)
1	Cadmium	39.163 + 2.445 = 41.608
2	Lead	27.519 + 1.176 = 28.695
3	Nickel	99.861 + 14.547 = 114.408
4	Zinc	69.697 + 23.832 = 93.529

Table 6.3 Limit of Detection for Cadmium, Lead, Zinc and Nickel for Edible oil sample by Stripping Voltammetry.

In the analysis for sample the table below summarized the result for Cadmium, Lead, Nickel and Zinc of the various edible oil products mainly are margarine, cooking oil and oil for salad dressing.

Sample Description	Cadmium (ug/L)	Lead (ug/L)	Nickel (ug/L)	Zinc (ug/L)
Palm oil Margarine (Bakery)	10.160	21.967	164.607	180.195
Palm oil Margarine	145.251	70.035	103.199	422.711
Sunflower Margarine	42.374	156.246	300.222	192.074
Corn Cooking Oil	14.079	9.401	138.891	No peak
Olive Oil	443.044	601.537	242.216	286.799
Sunflower Cooking Oil	173.065	65.663	61.381	429.058
Palm Cooking Oil	58.409	46.722	216.935	375.516

Table 6.4 Concentration (ug/L) of Nickel, Cadmium, lead and Zinc in various edible oil samples by Stripping Voltammetry

The result obtained in the table above is the concentration of the metal in diluted sample, therefore a calculation was performed to calculated the amount of the metal contain in per gram of sample. Since the sample after digestion was transferred and top up to 25ml in a volumetric flask, therefore the concentration of the metal in the sample is calculated by the followed formula:

$$\text{Concentration of Metal in sample (ug/g)} = \frac{(Cd - Cb)\ \text{ng/ml} \times 25\ \text{ml}}{\text{Sample saiz (g)} \times 1000}$$

whereby, Cd = Concentration of metal in diluted sample (ug/L)

Cb = Concentration of Metal in Reagent Blank (ug/L)

The table below summarized the concentration of the metal in sample.

Sample Description	**Sample Saiz (g)**	**Cd – Cb (ng/ml)**	**Concentration of Cadmium in ug/g**
Palm oil Margarine (Bakery)	1.4909	- 29.003	N.D
Palm oil Margarine	1.4971	106.088	1.772
Sunflower Margarine	1.4877	3.211	0.0540
Corn Cooking Oil	1.4366	-25.084	N.D
Olive Oil	1.5092	403.881	6.690
Sunflower Cooking Oil	1.5108	133.902	2.216
Palm Cooking Oil	1.4925	19.246	0.322

Table 6.5 Concentration (ug/g) of Cadmium in Edible oil samples

Sample Description	Sample Saiz (g)	Cd – Cb (ng/ml)	Concentration of Lead in ug/g
Palm oil Margarine (Bakery)	1.4909	-5.552	N.D
Palm oil Margarine	1.4971	42.516	0.710
Sunflower Margarine	1.4877	128.727	2.163
Corn Cooking Oil	1.4366	-18.118	N.D
Olive Oil	1.5092	574.018	9.509
Sunflower Cooking Oil	1.5108	38.144	0.631
Palm Cooking Oil	1.4925	19.203	0.322

Table 6.6 Concentration (ug/g) of Lead in Edible Oil Samples

Sample Description	Sample Saiz (g)	Cd – Cb (ng/ml)	Concentration of Nickel in ug/g
Palm oil Margarine (Bakery)	1.4909	64.746	1.619
Palm oil Margarine	1.4971	3.338	0.056
Sunflower Margarine	1.4877	200.361	3.367
Corn Cooking Oil	1.4366	39.030	0.679
Olive Oil	1.5092	142.355	2.358
Sunflower Cooking Oil	1.5108	- 38.48	N.D
Palm Cooking Oil	1.4925	117.074	1.961

Table 6.7 Concentration (ug/g) of Nickel in Edible Oil Samples

Sample Description	Sample Saiz (g)	Cd – Cb (ng/ml)	Concentration of Zinc in ug/g
Palm oil Margarine (Bakery)	1.4909	110.498	1.853
Palm oil Margarine	1.4971	353.014	5.895
Sunflower Margarine	1.4877	122.377	2.056
Corn Cooking Oil	1.4366	ND	N.D
Olive Oil	1.5092	217.102	3.596
Sunflower Cooking Oil	1.5108	359.361	5.947
Palm Cooking Oil	1.4925	305.819	5.123

Table 6.8 Concentration (ug/g) of Zinc in Edible Oil Samples

From the analysis result the Corn cooking oil (CCO) Sample does not contain any detectable amount of Lead, Zinc and Cadmium as the concentration of the particular metal is detect at below the blank value, therefore the concentration was reported as none detected (N.D), however the Nickel was detected in the Corn cooking oil sample at about 0.679 ug/g. From the voltammogram of lead and cadmium in the corn cooking oil analysis, both peaks were well separated with lead (Pb) peak position at -0.471 V and cadmium (Cd) at -0.477 V (Figure 6.9). The Concentration are higher for blank may be due to sample preparation is not well take care, as the sample container and digester vessel maybe contaminated by small amount of cadmium and lead, that might present in it since the apparatus is shared in the laboratory. Since the amount of blank is at a sub part per billion level therefore contamination is often difficult to be avoided unless proper cleaning of the vessel is well perform such as soaking with nitric acid solution, and

multiple rinsing with ultrapure water. For the cases of Zinc analysis in Corn Cooking Oil, the Zinc Peak was not well resolved and interferes by an unknown peak at position of -1.00 V (figure 6.9); as a result the Zinc peak was not detected and can't be quantified.

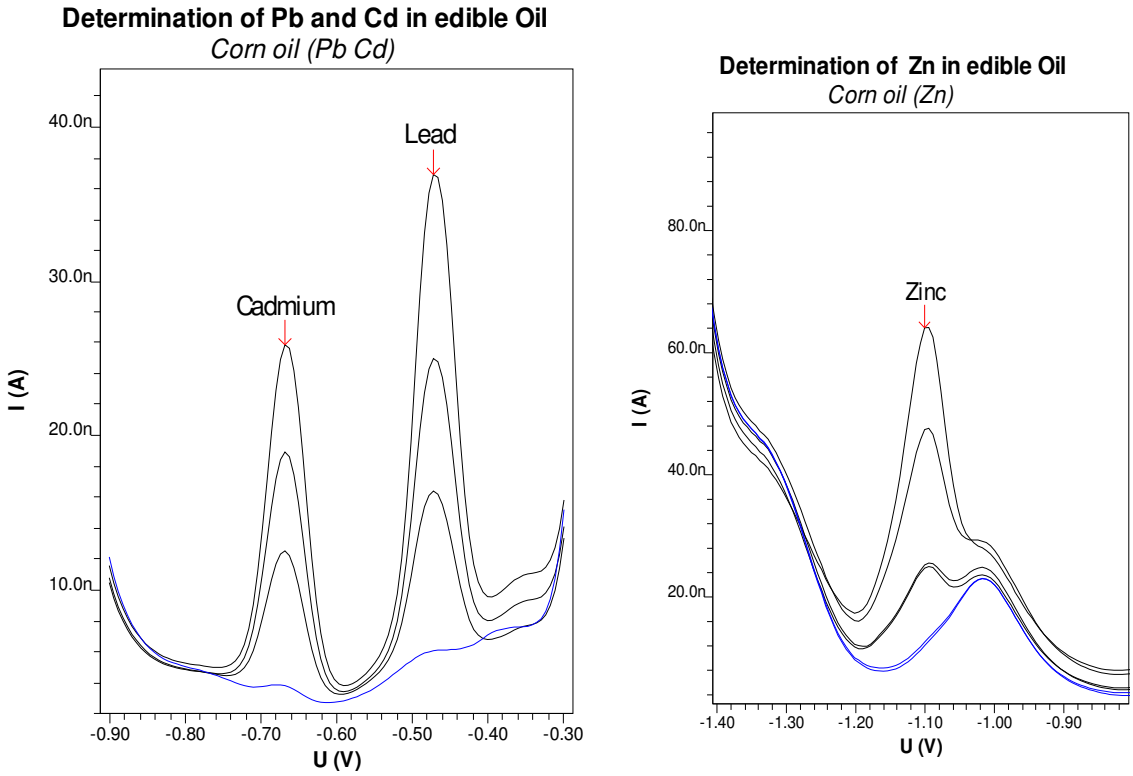


Figure 6.9 Voltammogram of Cadmium, Lead and Zinc in Corn Cooking Oil Sample.

The nickel peak for Corn cooking Oil is sharp and without any interference of baseline or other unknown peak (figure 6.10), the peak was positioned at -0.970 V.

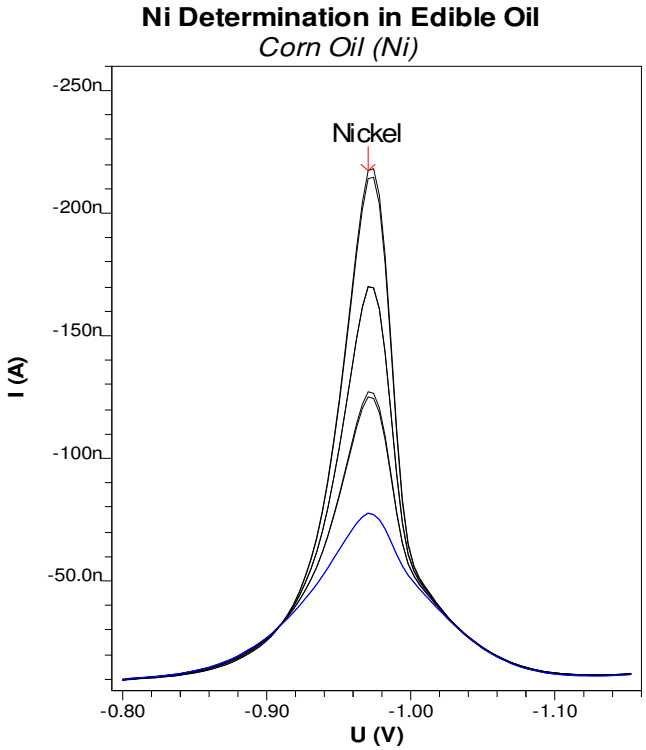


Figure 6.10 Voltammogram of Nickel in Corn Cooking Oil sample.

From the analysis result, the Olive oil sample was found to contain the highest concentration of Cadmium and Lead in comparison to other samples, with Cadmium contain about 6.697 ug/g and Lead 9.509 ug/g (figure 6.11). As in comparison to other sample the Lead (Pb) concentration was detected low for all samples except for Olive oil (9.509 ug/g) and sunflower margarine (2.163 ug/g). Whereas for Cadmium, it is detected high for Palm Oil Margarine (1.772 ug/g), Olive Oil (6.697 ug/g) and Sunflower Cooking Oil (2.216 ug/g). The detected value is far higher than those reported in literature as most is reported below than 1.0 ug/g. The high concentration of Lead and Cadmium in this sample is maybe possible due to contamination of microwave digester vessel and sample container.

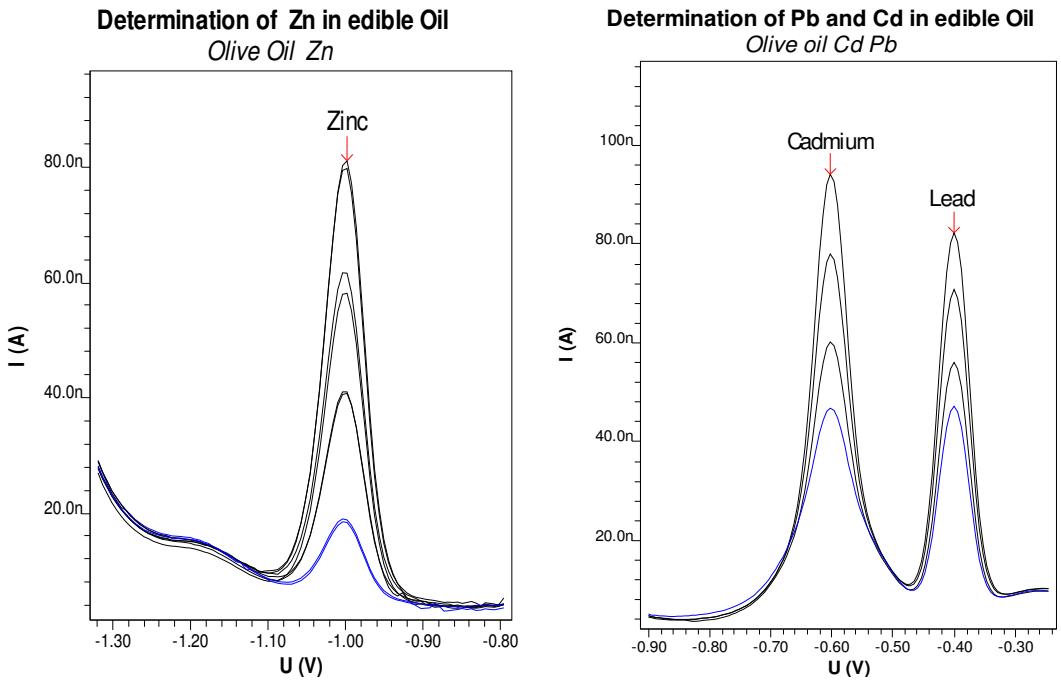


Figure 6.11, Voltammogram of Zinc, Cadmium and Lead in Olive Oil

As in the cases for Nickel the concentration detected in the sample is in the range of 0.250 to 3.500 ug/g which is inconsistent with the result that reported by the literature. This is similarly observed for the Zinc metal, as the range detected is in between 1.000 ug/g to 6.000 ug/g for all the sample and the result is consistent with the literature which reported at between 3.0 ug/g to 13 ug/g (Chapter 4.0).

For validation of the method, 3 spike samples were prepared and the recovery was determined to ensure the method reliability. The three sample selected was Olive Oil, Corn Oil, and Palm Oil Margarine (Bakery), an accurate spike of 100 ug/L of each Zinc, Nickel, Lead and cadmium was performed in the sample. The table summarized the result of the recovery studies.

Metal	Sample without spike ug/L	Sample with spike ug/L	Recovery (%)
Cadmium (Cd)	14.079	99.935	85.856
Lead (Pb)	9.401	106.313	96.912
Nickel (Ni)	138.891	237.016	98.125
Zinc (Zn)	No peak	101.450	101.450

Table 6.12 Recovery studies of spike 100 ug/L of Cadmium, Lead, Nickel and Zinc in Corn Oil Sample.

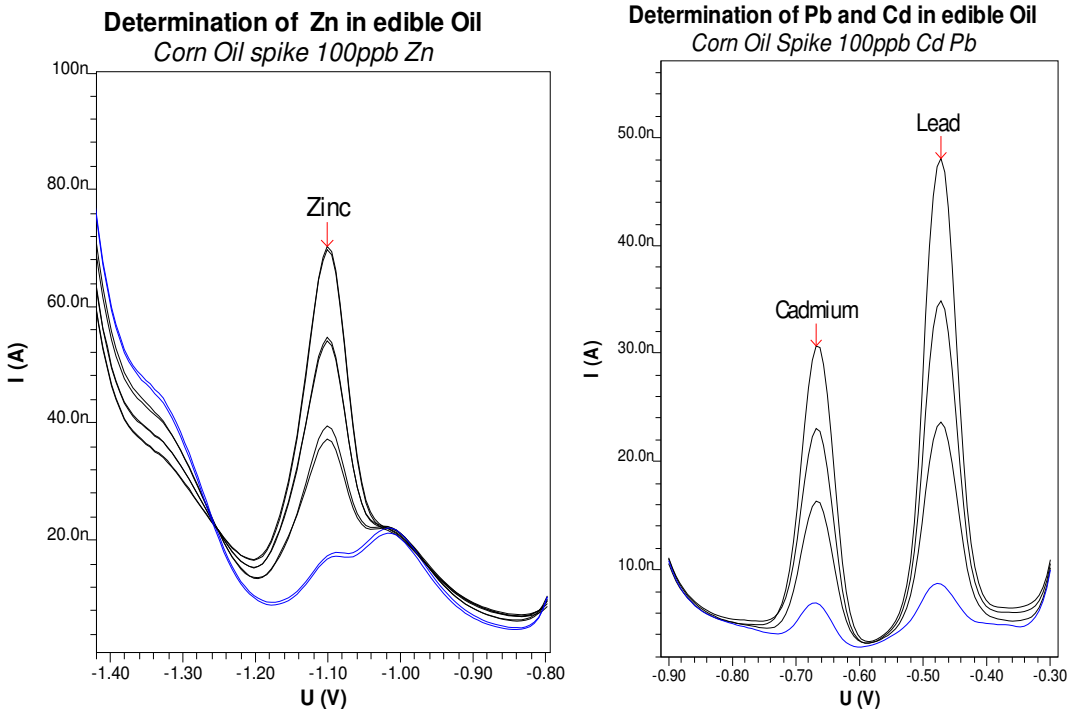


Figure 6.13 Voltammogram of spike Cadmium, Lead, and Zinc in Corn Oil Sample

The recovery studies of corn oil show that the Lead, Nickel and Zinc was recover at the range of 95% to 105%, which is acceptable except for Cadmium with recovery of 85.856% which is 14.144% less than expected. From the voltammogram, it can observe

that the signal of cadmium, Lead and zinc peak (figure 6.13) was increase in comparison to the un-spiked Corn Oil sample (figure 6.9).

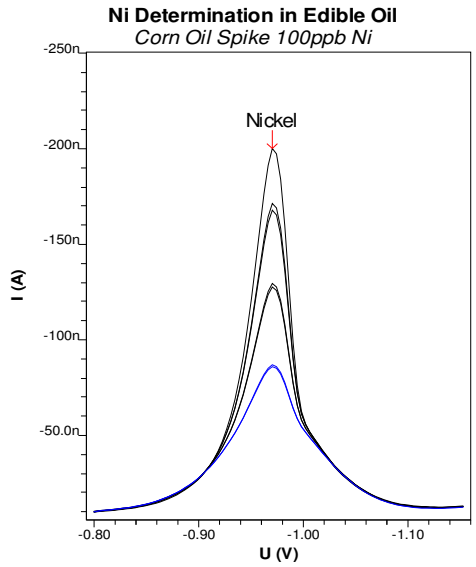


Figure 6.14 Voltammogram of spike Nickel in Corn Oil Sample

Metal	Sample without spike ug/L	Sample with spike ug/L	Recovery (%)
Cadmium (Cd)	443.044	544.882	101.838
Lead (Pb)	601.537	714.902	113.365
Nickel (Ni)	242.216	349.268	107.052
Zinc (Zn)	286.799	384.359	97.560

Table 6.15 Recovery studies of spike 100 ug/L of Cadmium, Lead, Nickel and Zinc in Olive Oil Sample.

The result of the olive oil spikes sample show a good recovery for Cadmium (101.838 %), Nickel (107.052 %) and Zinc (97.560 %) except for Lead (113.365 %) whereby the recovery was 13.365% more.

Metal	Sample without spike ug/L	Sample with spike ug/L	Recovery (%)
Cadmium (Cd)	10.160	91.567	81.407
Lead (Pb)	21.967	43.927	21.960
Nickel (Ni)	164.607	263.234	98.627
Zinc (Zn)	180.195	280.463	100.268

Table 6.16 Recovery studies of spike 100 ug/L of Cadmium, Lead, Nickel and Zinc in Palm Oil Margarine (Bakery) Sample.

For the cases of Palm oil margarine (bakery) spike sample it show only a good recovery for Nickel and Zinc with recovery of 98.627% and 100.268% respectively. However both Cadmium and Lead recovery was out from expectation value with lead only recovery by 21.960 % and Cadmium with 81.407 %.

From all the spike sample result, it shows that the Cadmium and Lead recovery was more than +/- 5%, the reason for this maybe because due to human error during calibration. In all three spike sample analysis, the volume of standard addition used for calibration in the cadmium and lead analysis was 50ul using micropipette. As in compare to Zinc and nickel, the volume of standard addition that used in the calibration was in the range of 100ul for Nickel and 250ul to 500ul for Zinc. The small volume used in standard addition for Cadmium and Lead maybe contribute to the error in the recovery studies, as 50ul standard addition maybe not accurate due to human error. Beside that, Lead and Cadmium was present in the sample at lower concentration of below 25ug/L in the sample in comparison to Nickel and Zinc, therefore the equipment sensitivity and contamination may result in the recovery error. Apart from that Lead are usually not

stable without acidify the spike sample; therefore the spike sample should be analyze freshly once prepared. However acidification of spike sample will affect the final pH value of the sample, which as a consequence will affect the peak resolution between Lead and Cadmium. The very low recovery of Lead in Palm Oil Margarine (bakery) sample maybe cause by the instability of lead or matrix problem in the sample, as the spike Palm oil Margarine sample preparation are not acidify.

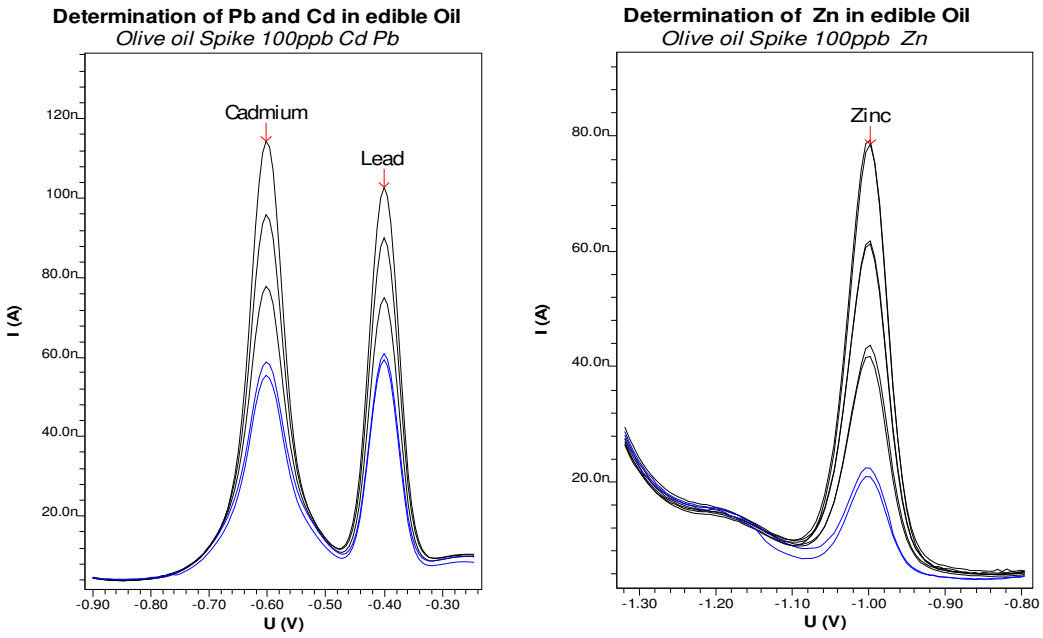


Figure 6.17 Voltammogram of spike Cadmium, Lead and Zinc in Olive Oil Sample

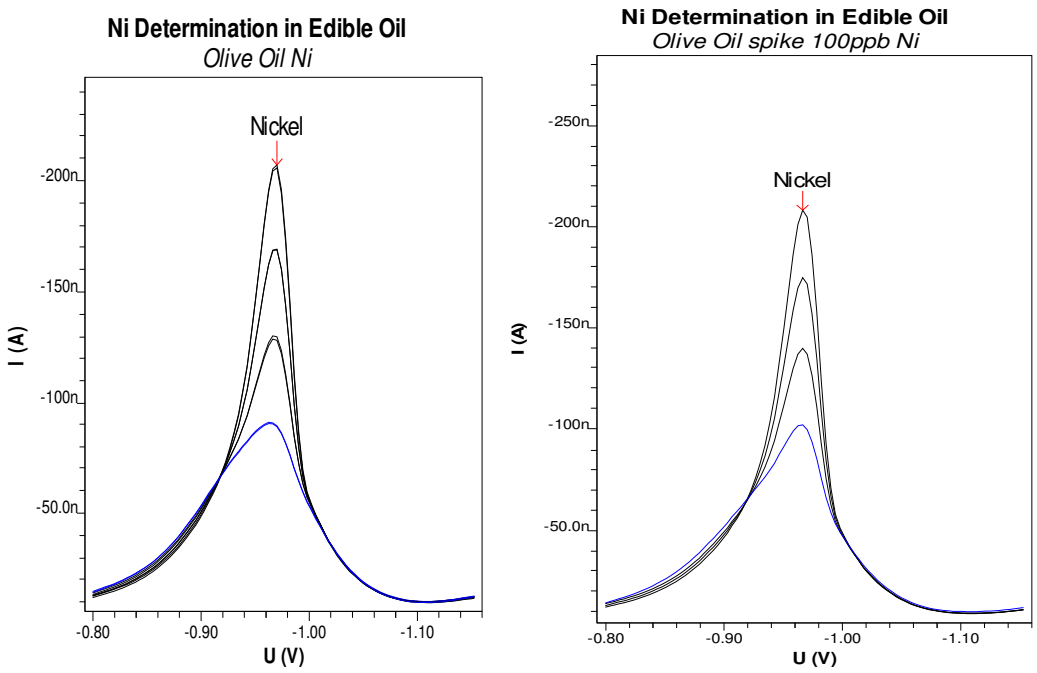


Figure 6.18 Voltammogram of Spike Nickel in Olive Oil Sample

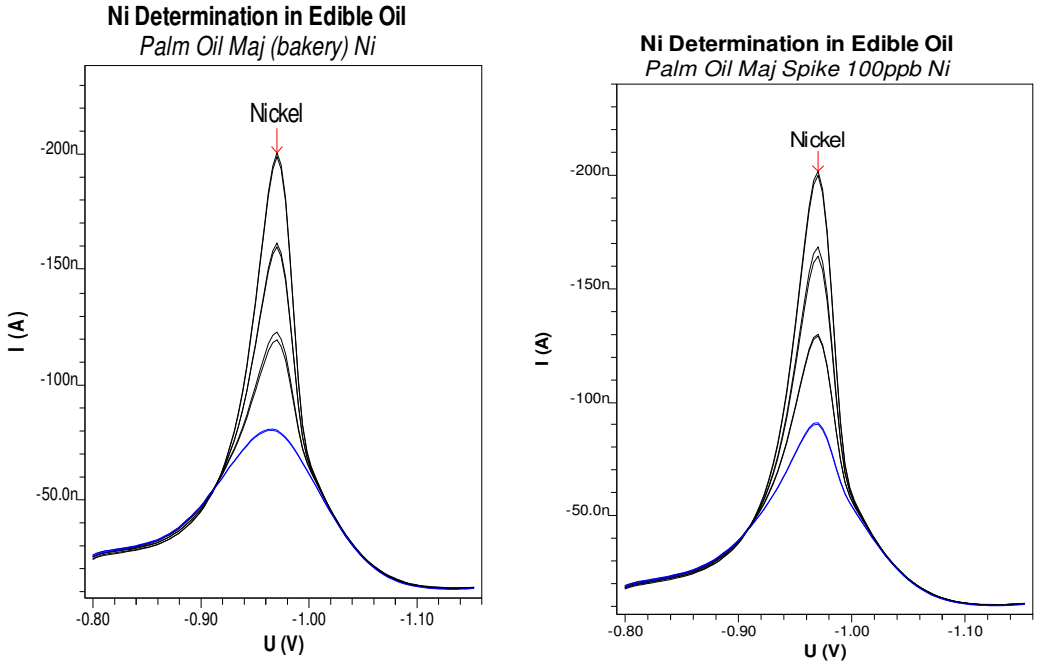


Figure 6.19 Voltammogram of Spike Nickel in Palm Oil Margarine (bakery) Sample

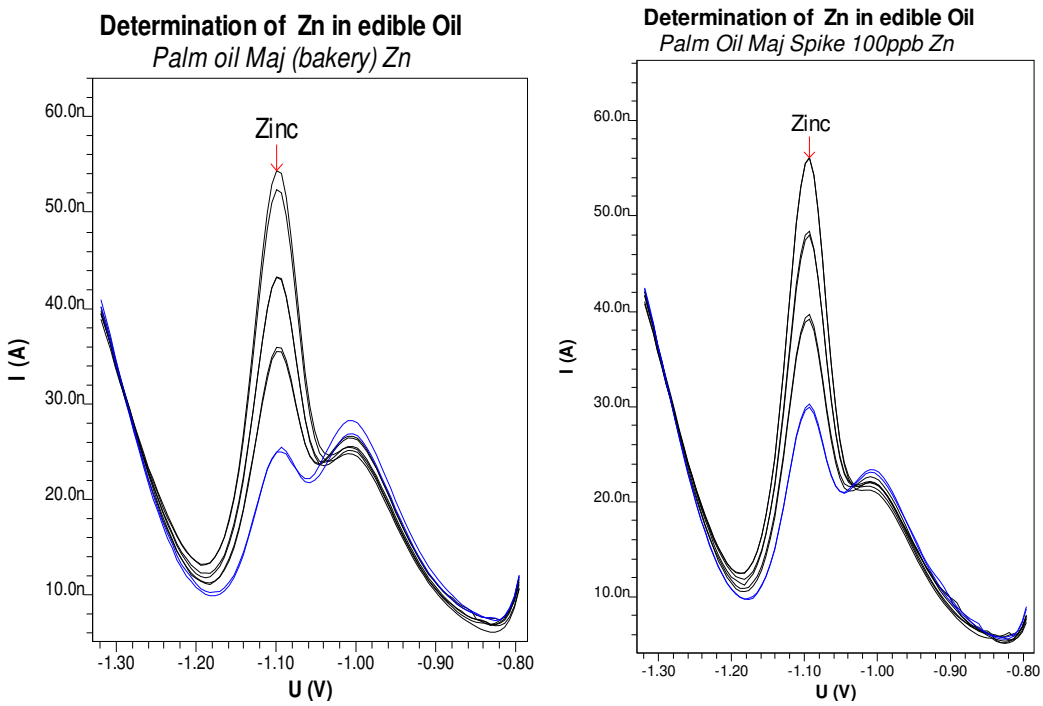


Figure 6.20 Voltammogram of Spike Zinc in Palm Oil Margarine (bakery) Sample

From the Voltammogram of Palm oil Based Margarine bakery (figure 6.18) it is clear that the Zinc signal increase sharply with spike sample, whereas the unknown peak remain the same peak height. The recovery of Zinc in this spike sample was 100.28%. In the analysis of metal by voltammetric in order for a peak to be well detected and quantify especially in simultaneous metal analysis the resolution of the peak voltage must be more than 100mv (F.G Thomas and G.henze, 2001), For the case of zinc in the palm oil sample it is clearly observed that the zinc peak is position at -1.12 v and unknown peak at -1.00 v, which mean both peak was separated by more than 100mv.

The Linear regression for each standard addition graph of the metal was calculated for each sample analysis, in order to study the linearity of the calibration by

standard addition method. The table below summarized the linear regression (R^2) for each of the calibration.

Sample description	Cadmium (Cd)	Lead (Pb)	Nickel (Ni)	Zinc (Zn)
Blank 1	0.9969	0.9983	0.9980	0.9978
Blank 2	0.9957	0.9934	0.9990	0.9941
Palm oil Margarine (Bakery)	0.9978	0.9957	1.0000	0.9983
Palm oil Margarine	0.9995	0.9994	0.9981	0.9995
Sunflower Margarine	0.9921	0.9919	0.9968	0.9988
Corn Cooking Oil	0.9965	0.9961	0.9997	0.9998
Olive Oil	0.9998	0.9994	0.9999	0.9995
Sunflower Cooking Oil	0.9981	0.9931	1.0000	0.9953
Palm Cooking Oil	0.9991	0.9935	0.9994	0.9911
Average	0.9973	0.9956	0.9990	0.9971

Table 6.21 Linear Regression (R^2) of the standard addition calibration graph for each metal

From the table, most of the sample analysis shows a linear regression of more than 0.999 except for some sample. In term of metal, nickel show a considerable good linearity with average of 0.9990, all the sample analyses with R^2 of more than 0.999 except for palm oil margarine and sunflower margarine sample, with R^2 of 0.9981 and 0.9968 respectively. For the case of Cadmium and Zinc the average linear regression for all the analysis was 0.997 above and for lead is 0.9956. The lower linear regression for cadmium and lead is expected, this is because the volume of standard addition used was very low in the range of 50 to 100ul, this maybe the possible contribution to the error since manual addition was performed. Whereas, For all the blank sample, most of the result have linear

regression of less than 0.999, this is because only 3 calibration point are used for the calibration therefore the data for the regression calculation maybe insufficient, beside that the standard addition was performed using a micropipette manually therefore error in addition maybe occurs.

7.0 CONCLUSION

As a conclusion, the analysis of Lead, Cadmium, Zinc and Nickel in this Edible oil product can be alternatively performed by Voltammetry Technique instead of other methods such as Atomic Absoprtion Spectroscopy (AAS), Graphite Furnace Atomic Absorption Spectroscopy (GFAAS) or Inductively Couple Plasma Mass Spectroscopy (ICP-MS). From this experiment, it shows that Voltammetry method can equally performed the metal analysis at the sub part per billion levels. Although the recovery and linearity in this experiment was not to it expectation with 100 +/- 5% recovery and calibration graph linear regression of more than 0.999, however further improvement can be performed by careful handling of sample preparation and it is highly recommended the use of high chemical grade such as Suprapure grade for preparation.

In Term of advantages, the Voltammetry technique is capable for simultaneous analysis of Zinc, Cadmium and Lead without required changing of working electrode or electrolyte. This is the advantage against AAS technique which is only single element analysis. In comparison to ICP-MS technique, the Voltammetry had a lower operational cost as it only uses nitrogen gas for purging and without any requirement for cooling of the system. The simple set up of voltammetry is suitable for quality control purposes as the equipment is easy handled and does not requires complicated settings. The only disadvantages of voltammetry technique is the used of mercury in working electrode, which often banned by many industries for its toxic handling.

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25) Cotton and Wilkinson. Advance Inorganic Chemistry. John Wiley and sons, Fifth edition, 1998.

APPENDIX 1

========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : BLk Cd Pb dth
Sample ID : BLk Cd Pb
Creator determ.: Date : 2009-12-23 Time: 10:35:41
Modified by : Date : 2010-01-21 Time: 23:35:26

Method : Determination of Cd and Pb in edible Oil with Acetate buffer
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.5 smpl + 10ml UPW)+0.25ml acetate buffer
Remark2 : deposition time 30s

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 50.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.692	2.12	2.20	0.070	0.00	
1 - 2	-0.692	2.23				
1 - 3	-0.692	2.26				
2 - 1	-0.692	9.75	8.95	0.729	6.74	
2 - 2	-0.692	8.76				
2 - 3	-0.692	8.33				
3 - 1	-0.692	15.21	14.48	0.784	5.53	
3 - 2	-0.692	14.58				
3 - 3	-0.692	13.65				

Substance : Lead
Conc. : 0.500 mg/L
Conc.dev. : ---
Add.amount : 50.000 ul

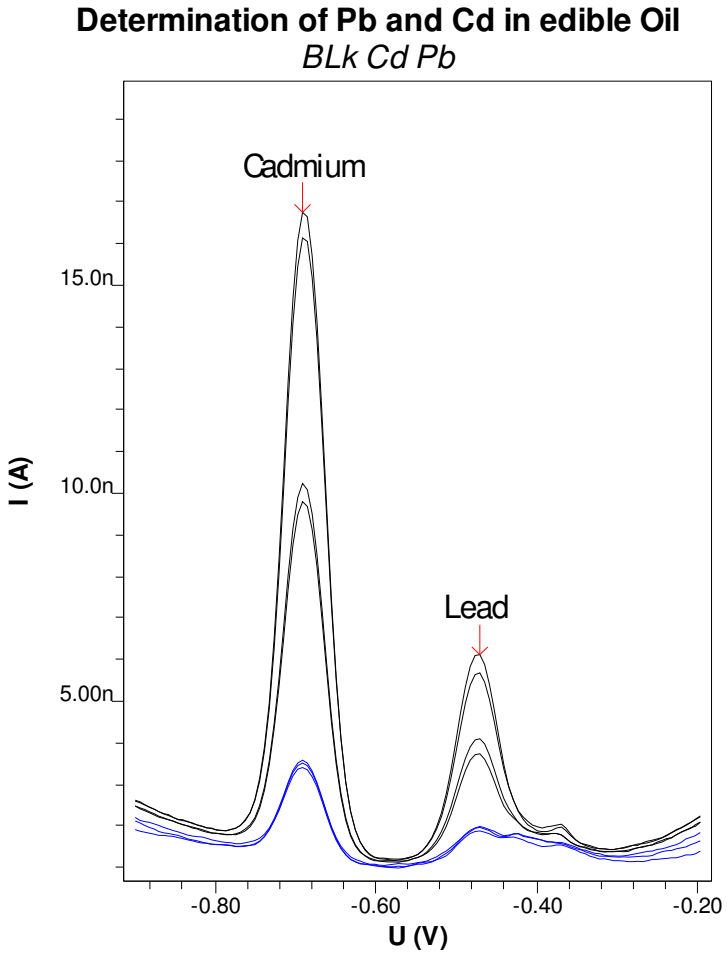
VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.471	0.841	0.848	0.014	0.000	
1 - 2	-0.471	0.863				
1 - 3	-0.471	0.839				
2 - 1	-0.471	3.393	2.915	0.449	2.067	
2 - 2	-0.471	2.852				
2 - 3	-0.471	2.501				
3 - 1	-0.471	4.838	4.317	0.555	1.402	

3 - 2 -0.471 4.382
3 - 3 -0.471 3.732

Solutions

No.	Content	Predose (mL)

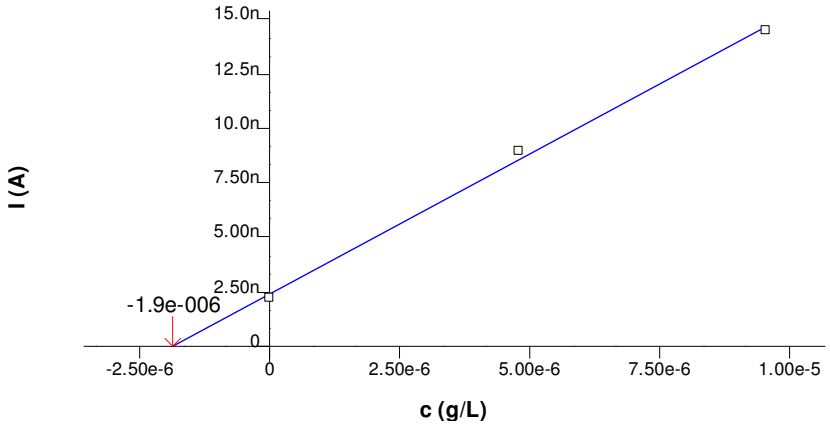
Final results			+/- Res. dev.	%	Comments
Cadmium:					
default	=	38.586 ug/L	5.835	15.121	
Lead :					
default	=	27.242 ug/L	7.068	25.947	



Cadmium

c = 38.586 ug/L

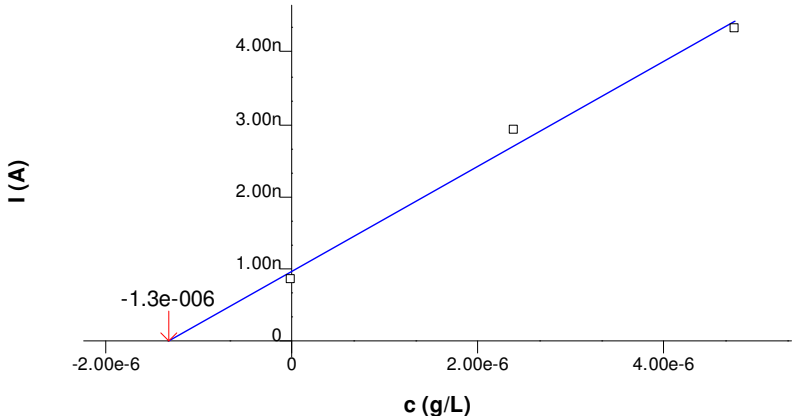
+/- 5.835 ug/L (15.12%)



Lead

c = 27.242 ug/L

+/- 7.068 ug/L (25.95%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : BLk Ni.dth
Sample ID : Blk Ni 1
Creator determ.: Date : 2009-12-23 Time: 11:24:51
Modified by : Date : 2010-01-22 Time: 00:09:20

Method : Ni Determination in Edible Oil (continous method).mth
Title : Determination of Ni in edible oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : Deposition time 5s, total volume 11.4

Sample amount : 0.500 mL
Cell volume : 11.400 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.994	-16.39	-16.48	0.110	0.00	
1 - 2	-0.994	-16.61				
1 - 3	-0.994	-16.44				
2 - 1	-0.994	-32.56	-33.20	0.773	-16.72	
2 - 2	-0.994	-32.97				
2 - 3	-0.994	-34.06				
3 - 1	-0.994	-49.28	-47.49	1.601	-14.29	
3 - 2	-0.994	-46.20				
3 - 3	-0.994	-46.99				

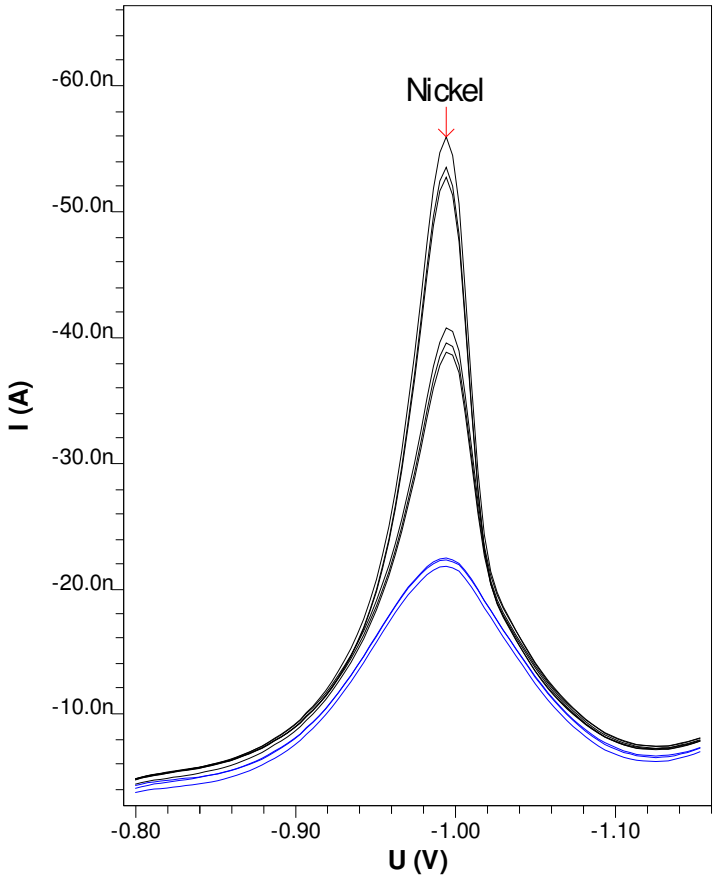
Solutions

No.	Content	Predose (mL)

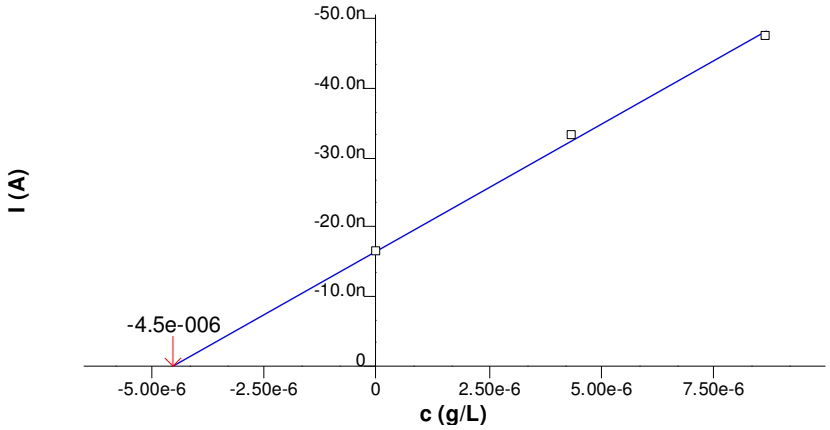
Final results	+/-	Res. dev.	%	Comments
Nickel:				
default =	103.289 ug/L	3.138	3.038	

Determination of Ni in edible oil

Blk Ni 1



Nickel
c = 103.289 ug/L
+/- 3.138 ug/L (3.04%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : BLk Zn.dth
Sample ID : BLk Zn 1
Creator determ.: Date : 2009-12-23 Time: 10:59:53
Modified by : Date : 2010-01-21 Time: 23:54:03

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl continous from previous Cd pb)
Remark2 : Deposition time 40s, total volume 10.50. stiring 1000rpm

---

Sample amount : 0.500 mL
Cell volume : 10.500 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 50.000ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-1.101	1.500	1.446	0.051	0.000	
1 - 2	-1.101	1.440				
1 - 3	-1.101	1.399				
2 - 1	-1.101	3.677	3.575	0.134	2.128	
2 - 2	-1.101	3.624				
2 - 3	-1.101	3.423				
3 - 1	-1.101	5.332	5.374	0.063	1.799	
3 - 2	-1.101	5.344				
3 - 3	-1.101	5.446				

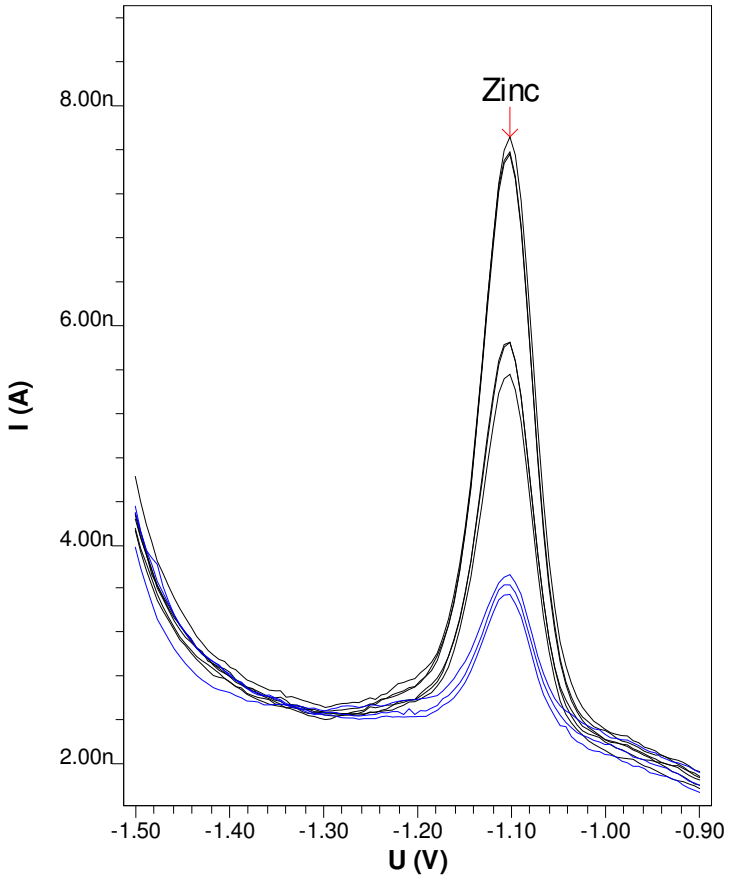
Solutions

No.	Content	Predose (mL)

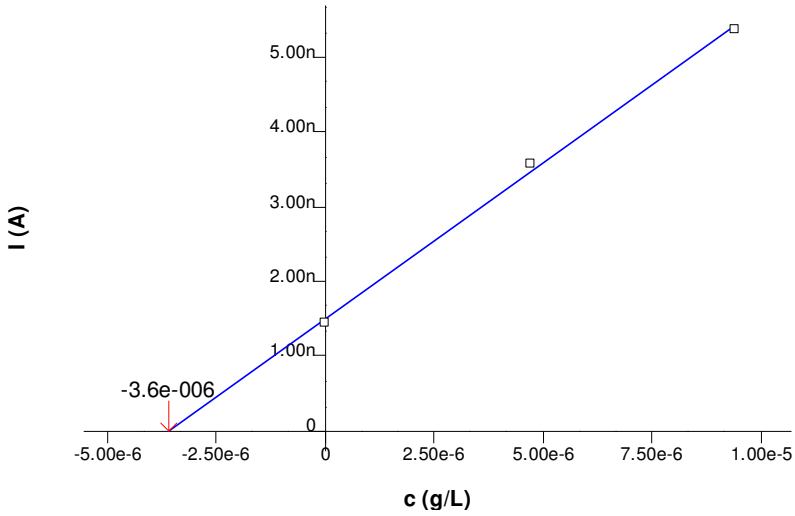
Final results	+/-	Res. dev.	%	Comments
Zinc:				
default =	75.314 ug/L	4.375	5.809	

Determination of Zn in edible Oil

BLk Zn 1



Zinc
c = 75.314 ug/L
+/- 4.375 ug/L (5.81%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : BLk Pb Cd 2 .dth
Sample ID : BLk 2 Pb Cd
Creator determ.: Date : 2009-12-23 Time: 11:56:22
Modified by : Date : 2010-01-21 Time: 23:36:37

Method : Determination of Cd and Pb in edible Oil with Acetate buffer
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.1 smpl + 10ml UPW)+0.25ml acetate buffer
Remark2 : deposition time 30s

Sample amount : 0.100 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 50.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.692	2.32	2.37	0.050	0.00	
1 - 2	-0.692	2.35				
1 - 3	-0.692	2.42				
2 - 1	-0.692	9.94	9.60	0.359	7.24	
2 - 2	-0.692	9.65				
2 - 3	-0.692	9.22				
3 - 1	-0.692	15.66	15.32	0.483	5.72	
3 - 2	-0.692	14.98				
3 - 3	-0.692	14.06				

Substance : Lead
Conc. : 0.500 mg/L
Conc.dev. : ---
Add.amount : 50.000 ul

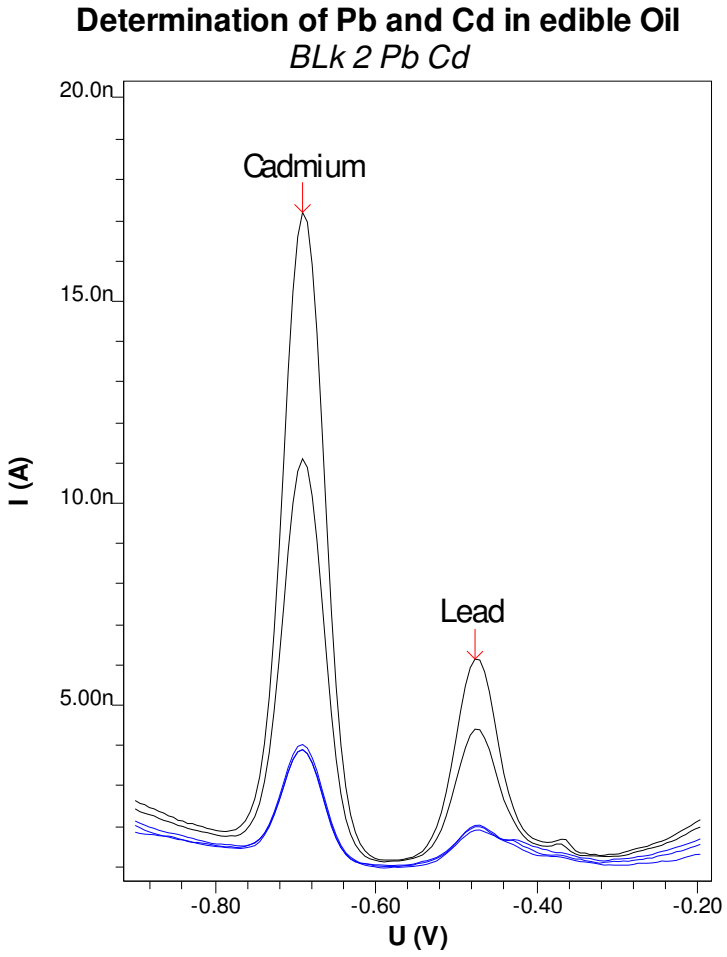
VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.471	0.904	0.934	0.026	0.000	
1 - 2	-0.477	0.947				
1 - 3	-0.471	0.950				
2 - 1	-0.477	3.445	3.176	0.294	2.242	
2 - 2	-0.477	3.221				
2 - 3	-0.477	2.862				
3 - 1	-0.477	4.927	4.681	0.347	1.505	

3 - 2 -0.477 4.436
3 - 3 -0.477 3.873

Solutions

No.	Content	Predose (mL)

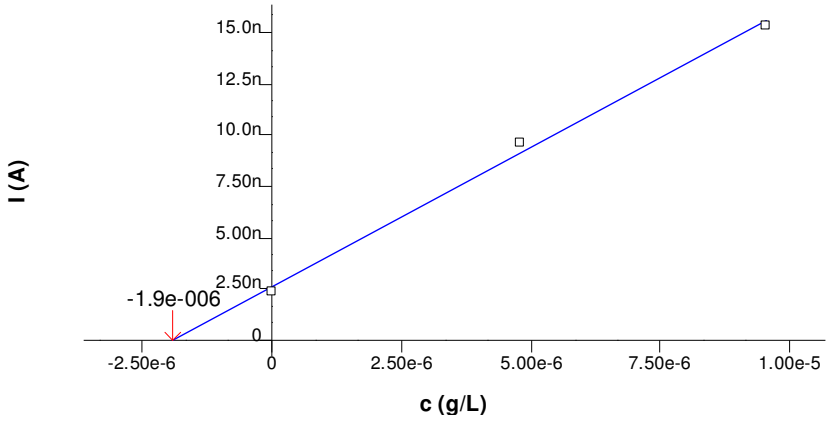
Final results			+/- Res. dev.	%	Comments
Cadmium:					
default	=	39.739 ug/L	4.901	12.334	
Lead :					
default	=	27.796 ug/L	5.285	19.012	



Cadmium

c = 39.739 ug/L

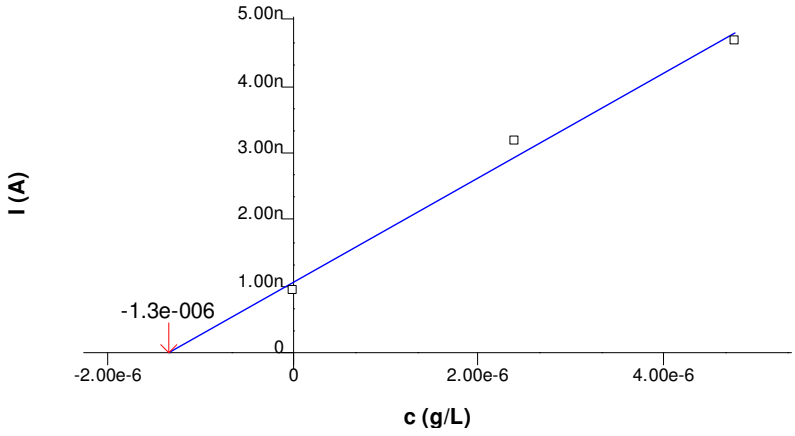
+/- 4.901 ug/L (12.33%)



Lead

c = 27.796 ug/L

+/- 5.285 ug/L (19.01%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : BLK Ni.dth
Sample ID : Blk 2 Ni
Creator determ.: Date : 2009-12-23 Time: 12:50:50
Modified by : Date : 2010-01-22 Time: 00:11:52

Method : Ni Determination in Edible Oil (continous method).mth
Title : Determination of Ni in edible oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : Deposition time 5s, total volume 11.4

Sample amount : 0.500 mL
Cell volume : 11.400 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.994	-15.71	-15.63	0.186	0.00	
1 - 2	-0.994	-15.77				
1 - 3	-0.994	-15.42				
2 - 1	-0.994	-32.20	-32.43	0.550	-16.79	
2 - 2	-0.994	-32.03				
2 - 3	-0.994	-33.06				
3 - 1	-0.994	-48.52	-47.22	1.243	-14.80	
3 - 2	-0.994	-47.12				
3 - 3	-0.994	-46.04				

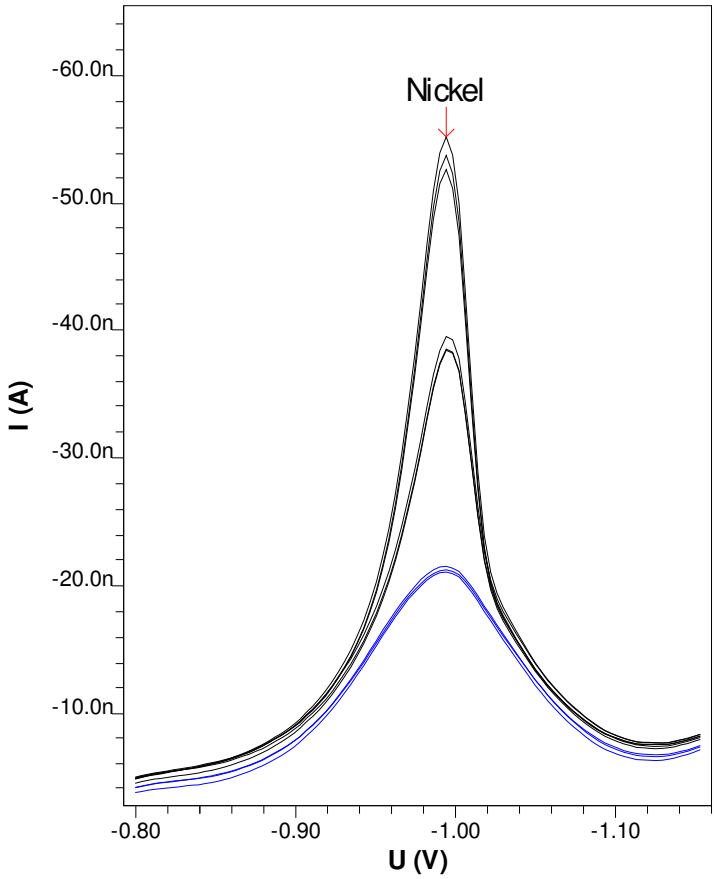
Solutions

No.	Content	Predose (mL)

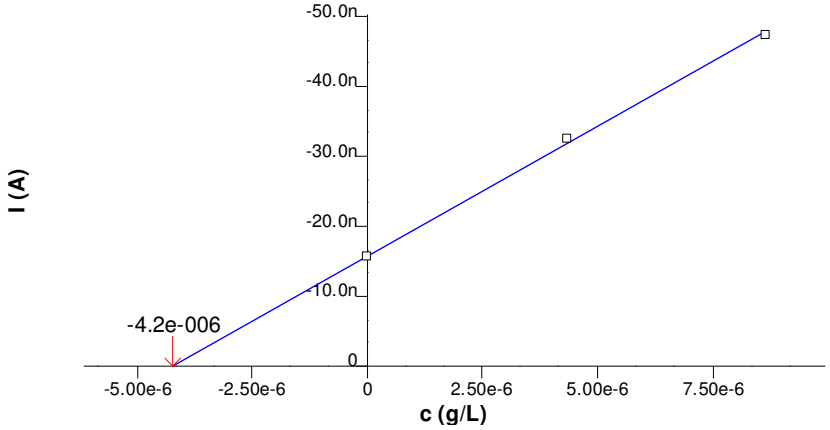
Final results	+/-	Res. dev.	%	Comments
Nickel:				
default =	96.432 ug/L	2.343	2.429	

Ni Determination in Edible Oil

Blk 2 Ni



Nickel
c = 96.432 ug/L
+/- 2.343 ug/L (2.43%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : BLk Zn.dth
Sample ID : BLk 2 Zn
Creator determ.: Date : 2009-12-23 Time: 12:28:23
Modified by : Date : 2010-01-21 Time: 23:55:10

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl continous from previous Cd pb)
Remark2 : Deposition time 40s, total volume 10.50. stiring 1000rpm

---

Sample amount : 0.500 mL
Cell volume : 10.500 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 50.000ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-1.101	1.139	1.123	0.014	0.000	
1 - 2	-1.101	1.113				
1 - 3	-1.101	1.117				
2 - 1	-1.101	3.377	3.231	0.143	2.108	
2 - 2	-1.101	3.225				
2 - 3	-1.101	3.091				
3 - 1	-1.101	4.970	4.836	0.121	1.605	
3 - 2	-1.101	4.801				
3 - 3	-1.101	4.736				

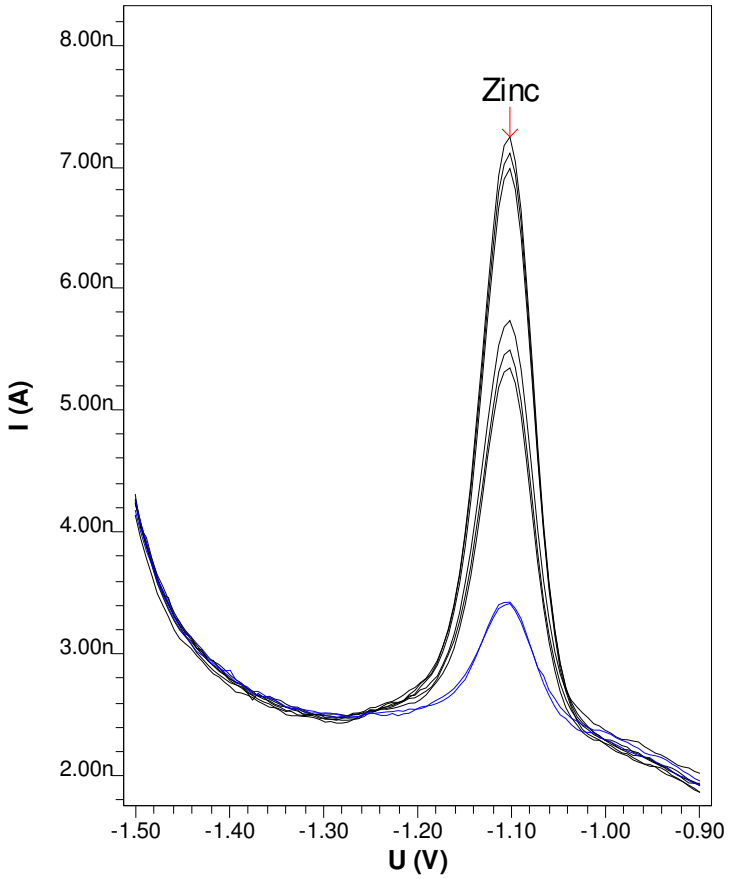
Solutions

No.	Content	Predose (mL)

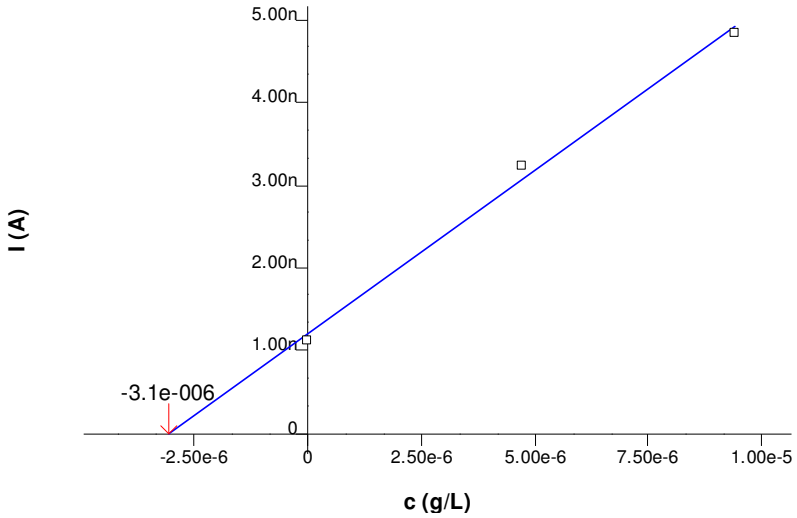
Final results	+/- Res. dev.	%	Comments
Zinc:			
default = 64.080 ug/L	6.376	9.950	

Determination of Zn in edible Oil

BLk 2 Zn



Zinc
c = 64.080 ug/L
+/- 6.376 ug/L (9.95%)



======== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Corn oil (Pb Cd).dth
Sample ID : Corn oil (Pb Cd)
Creator determ. : Date : 2009-12-30 Time: 16:05:02
Modified by : Date : 2010-01-20 Time: 14:20:42

Method : Determination of Cd and Pb in edible Oil with Acetate buffer
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.500 smpl + 10ml UPW)+0.25 Acetate buffer buffer
Remark2 : Deposition time 30 s

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 50.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.668	0.56	0.56	0.003	0.00	
1 - 2	-0.668	0.56				
2 - 1	-0.668	9.66	8.69	---	8.13	
2 - 2	-0.668	8.69				
3 - 1	-0.668	17.18	14.98	---	6.29	
3 - 2	-0.668	14.98				
4 - 1	-0.668	21.46	21.46	---	6.48	
4 - 2	-0.668	19.39				

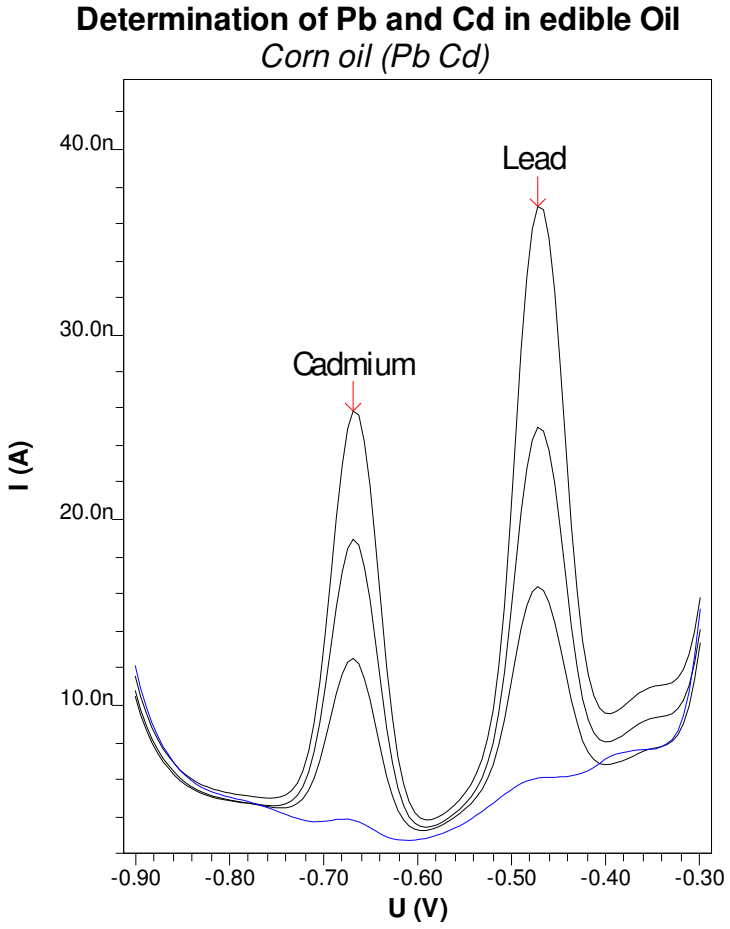
Substance : Lead
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 50.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	---	---	0.66	---	0.00	
1 - 2	-0.477	0.66				
2 - 1	-0.471	13.91	11.01	---	10.35	
2 - 2	-0.471	11.01				
3 - 1	-0.471	24.46	18.80	---	7.79	
3 - 2	-0.471	18.80				
4 - 1	-0.471	29.78	29.78	---	10.98	
4 - 2	-0.471	23.49				

Solutions

No.	Content	Predose (mL)
1	Standard For Cd and Pb	-------

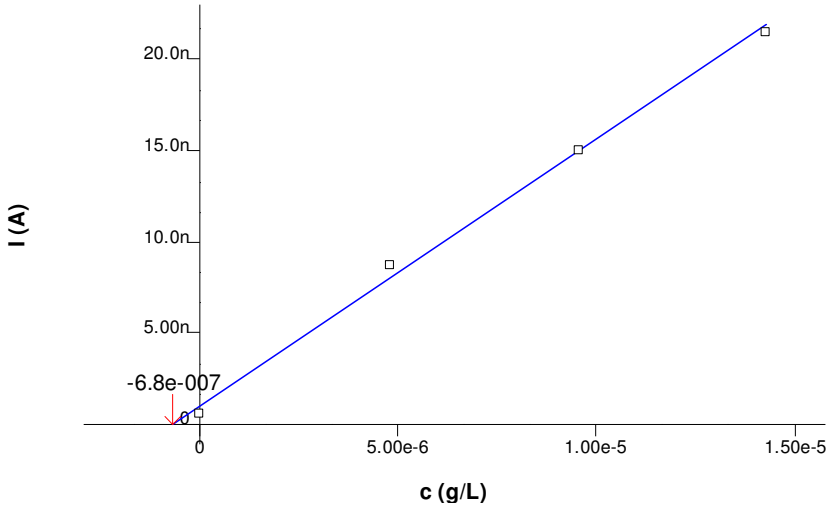
Final results	+/- Res. dev.	%	Comments
Cadmium:			
default = 14.079 ug/L	4.905	34.840	
Lead :			
default = 9.401 ug/L	4.668	49.656	



Cadmium

c = 14.079 ug/L

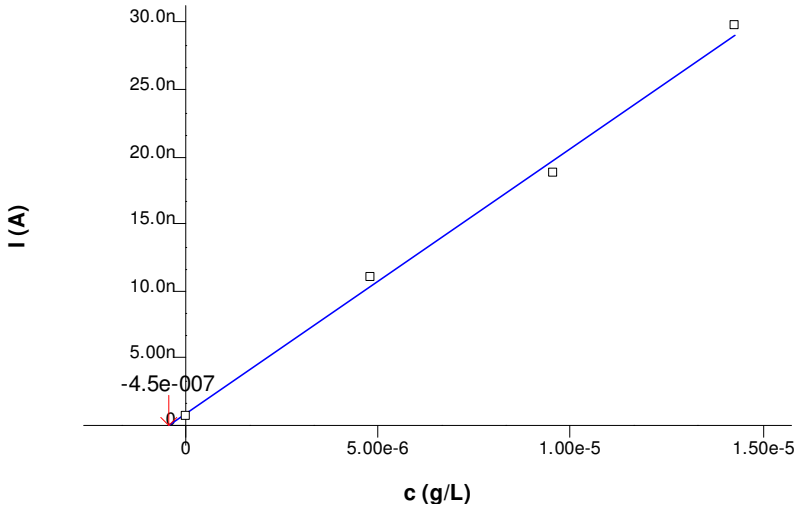
+/- 4.905 ug/L (34.84%)



Lead

c = 9.401 ug/L

+/- 4.668 ug/L (49.66%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Corn Oil (Ni).dth
Sample ID : Corn Oil (Ni)
Creator determ.: Date : 2009-12-30 Time: 16:37:09
Modified by : Date : 2010-01-20 Time: 15:44:57

Method : Ni Determination in Edible Oil (continous method).mth
Title : Ni Determination in Edible Oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : Deposition time 5s, total volume 14.75

Sample amount : 0.500 mL
Cell volume : 14.750 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.970	-30.5	-30.5	0.094	0.0	
1 - 2	-0.970	-30.6				
2 - 1	-0.970	-74.5	-75.5	1.498	-45.0	
2 - 2	-0.970	-76.6				
3 - 1	-0.970	-116.2	-116.4	0.311	-40.9	
3 - 2	-0.970	-116.7				
4 - 1	-0.970	-160.6	-159.2	1.924	-42.8	
4 - 2	-0.970	-157.8				

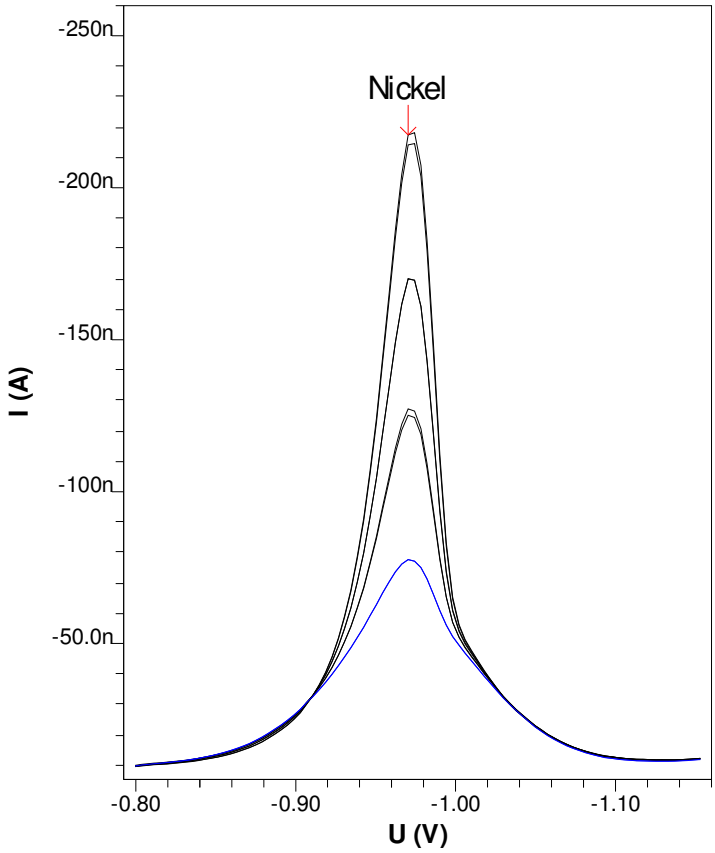
Solutions

No.	Content	Predose (mL)

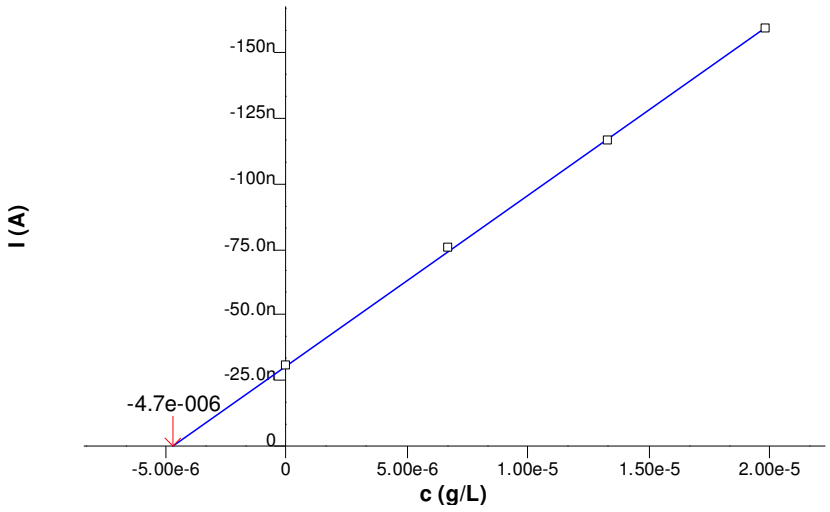
Final results	+/-	Res. dev.	%	Comments
Nickel:				
default = 138.891 ug/L	2.439		1.756	

Ni Determination in Edible Oil

Corn Oil (Ni)



Nickel
c = 138.891 ug/L
+/- 2.439 ug/L (1.76%)



======== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination :Corn oil (Zn) dth
Sample ID : Corn oil (Zn)
Creator determ.: Date : 2009-12-30 Time: 15:59:17
Modified by : Date : 2010-01-20 Time: 16:05:39

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl continous from previous Cd pb)
Remark2 : Deposition time 40s, total volume 10.65. stiring 1000rpm

---

Sample amount : 0.500 mL
Cell volume : 10.650 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : Variable. Add 1 - 0.500ml, Add 2 – 1.000ml, Add 3-1.000ml

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	---	---	---	---		
1 - 2	---	---				
2 - 1	-1.101	6.78	6.66	0.171	6.66	
2 - 2	-1.101	6.54				
3 - 1	-1.101	22.34	22.74	0.563	16.08	
3 - 2	-1.101	23.14				
4 - 1	-1.101	37.38	36.74	0.910	14.00	
4 - 2	-1.101	36.10				

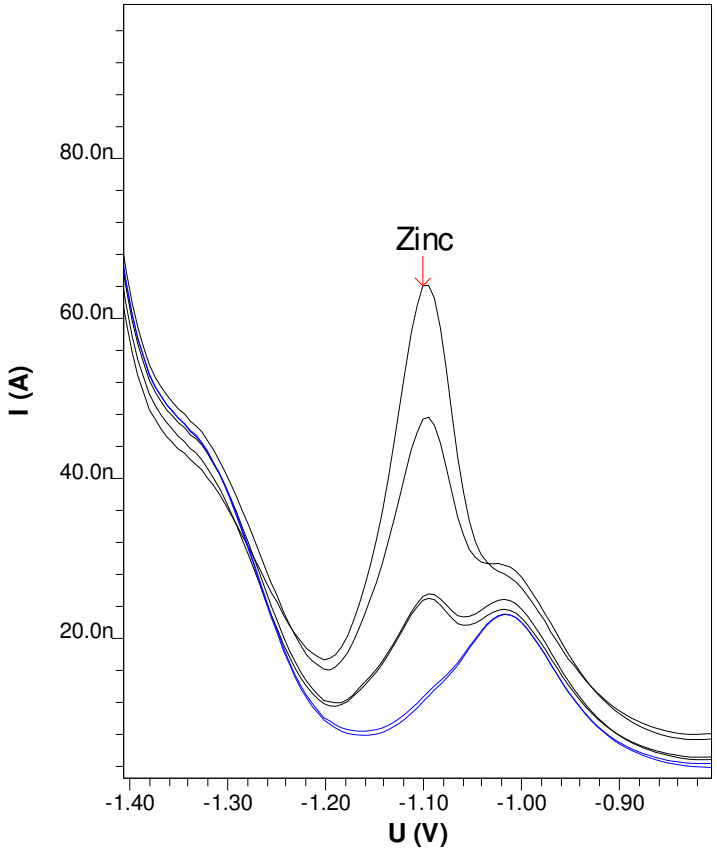
Solutions

No.	Content	Predose (mL)

Final results	+/- Res. dev.	%	Comments
Zinc:			
default = --- mg/L			No result found

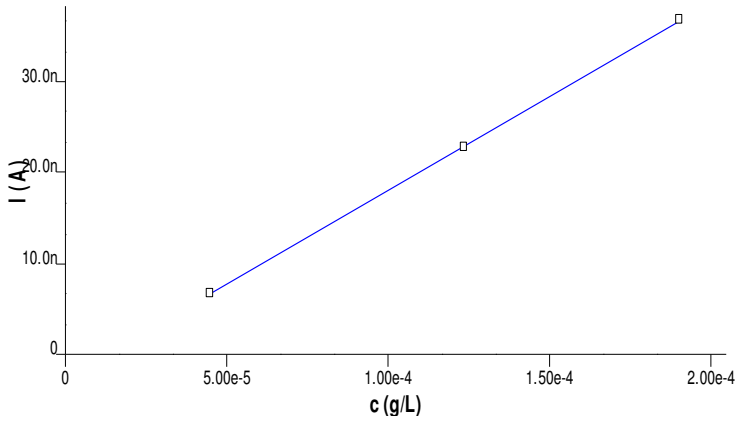
Determination of Zn in edible Oil

Corn oil (Zn)



Zinc

c = ---



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Corn Oil Spike 100ppb Cd Pb.dth
Sample ID : Corn Oil Spike 100ppb (Pb Cd)
Creator determ.: Date : 2010-01-02 Time: 13:20:27
Modified by : Date : 2010-01-20 Time: 14:46:12

Method : Determination of Cd and Pb in edible Oil with Acetate Buffer
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.50 smpl + 10ml UPW)+0.25 acetate buffer
Remark2 : Deposition time 30 s

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. Std : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.668	3.75	3.61	0.196	0.00	
1 - 2	-0.668	3.47				
2 - 1	-0.668	12.76	12.61	0.208	9.00	
2 - 2	-0.668	12.46				
3 - 1	-0.668	20.84	19.94	1.270	7.33	
3 - 2	-0.668	19.05				
4 - 1	-0.668	26.69	26.69		6.75	
4 - 2	-0.662	24.08				

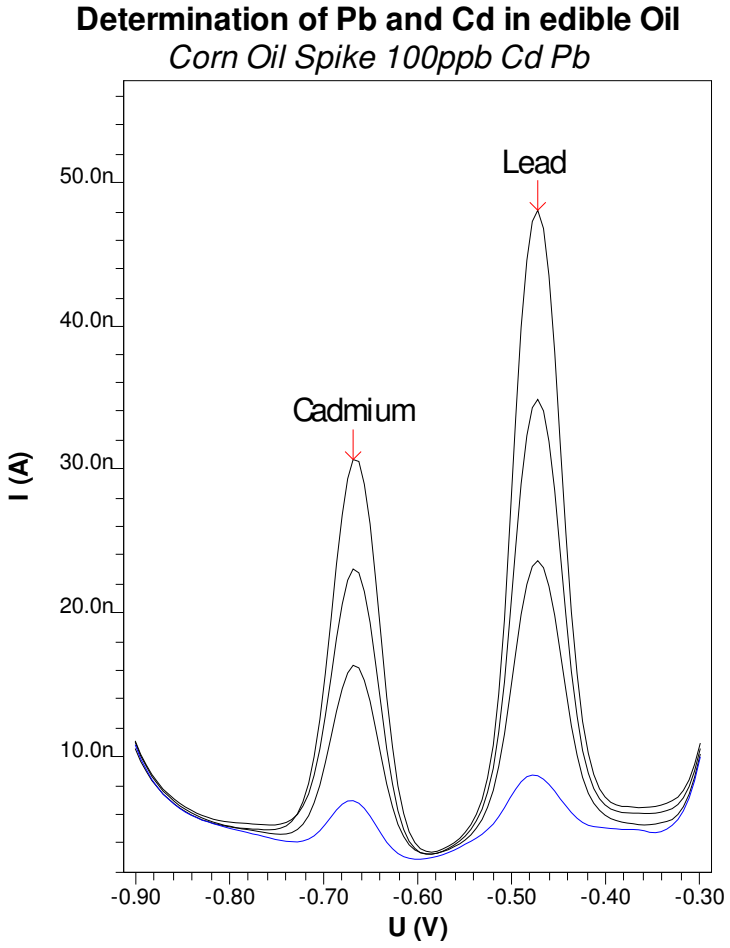
Substance : Lead
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.477	8.64	6.61	2.865	0.00	
1 - 2	-0.477	4.59				
2 - 1	-0.471	21.99	20.72	1.793	14.11	
2 - 2	-0.471	19.45				
3 - 1	-0.471	34.89	32.62	3.208	11.91	
3 - 2	-0.471	30.36				
4 - 1	-0.471	43.24	43.24	---	10.62	
4 - 2	-0.471	37.80				

Solutions

No.	Content	Predose (mL)
1	Standard For Cd and Pb	-------

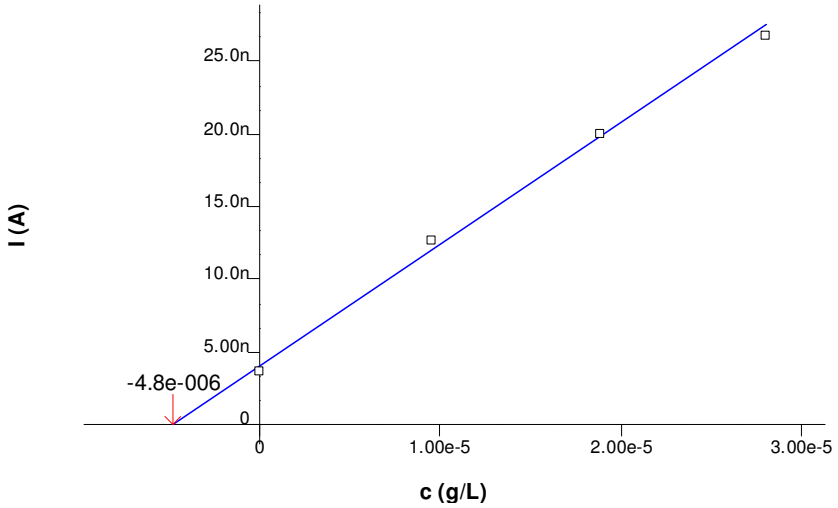
Final results	+/-	Res. dev.	%	Comments
Cadmium:				
default = 99.935 ug/L		13.714	13.723	
Lead :				
default = 106.313 ug/L		22.289	20.965	



Cadmium

c = 99.935 ug/L

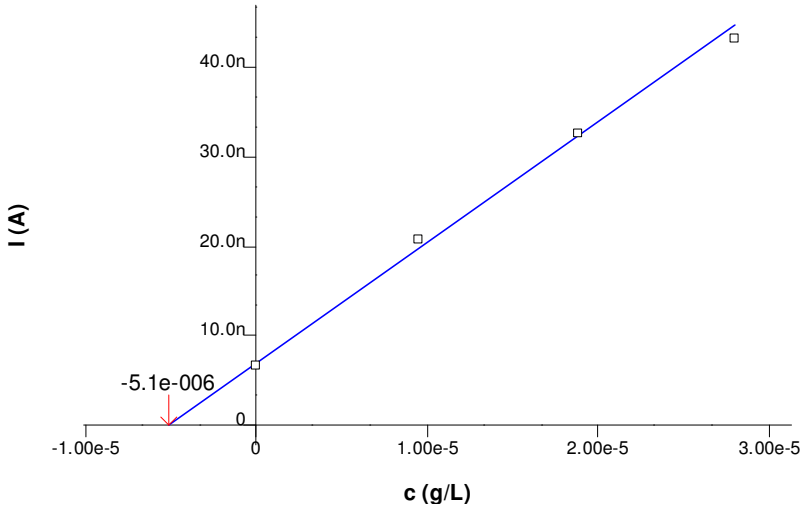
+/- 13.714 ug/L (13.72%)



Lead

c = 106.313 ug/L

+/- 22.289 ug/L (20.97%)



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination :Corn Oil Spike 100ppb Ni.dth
Sample ID : Corn Oil Spike 100ppb Ni
Creator determ.: Date : 2010-01-02 Time: 14:20:13
Modified by : Date : 2010-01-20 Time: 15:44:30

Method : Ni Determination in Edible Oil (continous method).mth
Title : Ni Determination in Edible Oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : deposition time 5s, total vol 14.75

Sample amount : 0.500 mL
Cell volume : 14.750 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.970	-47.1	-46.7	0.639	0.0	
1 - 2	-0.970	-46.2				
2 - 1	-0.970	-88.5	-87.7	1.199	-41.0	
2 - 2	-0.970	-86.8				
3 - 1	-0.970	-129.7	-127.8	2.622	-40.2	
3 - 2	-0.970	-126.0				
4 - 1	-0.970	-168.2	-158.3	---	-30.5	
4 - 2	-0.970	-158.3				

Solutions

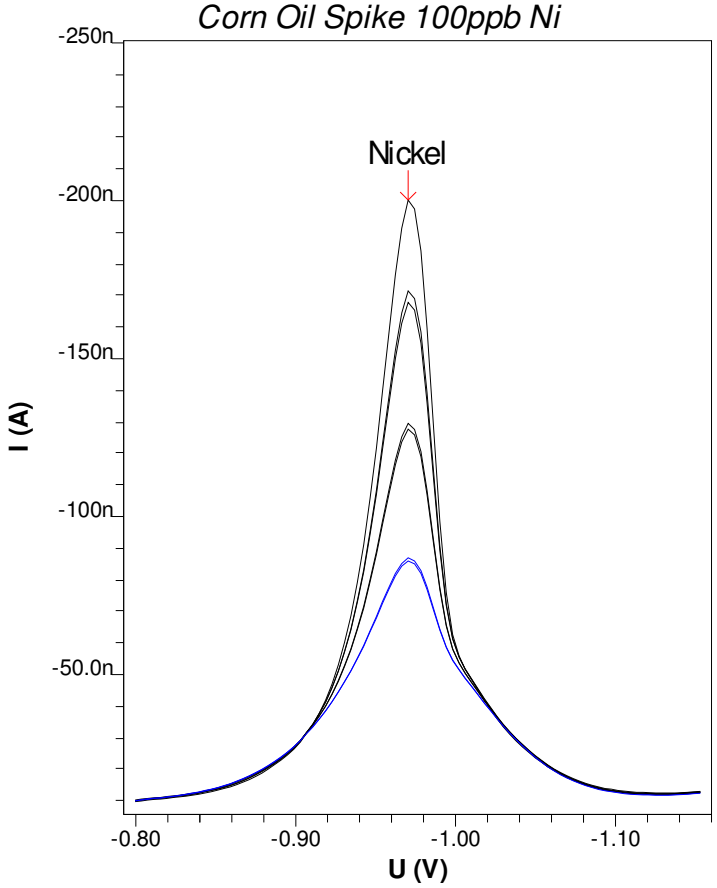
No.	Content	Predose (mL)

Final results	+/-	Res. dev.	%	Comments

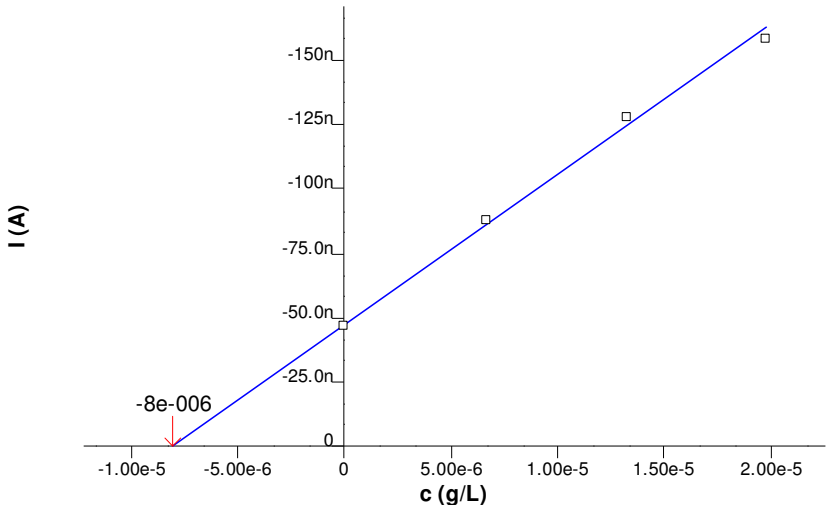
Nickel:
default = 237.016 ug/L 8.743 3.689

Ni Determination in Edible Oil

Corn Oil Spike 100ppb Ni



Nickel
c = 237.016 ug/L
+/- 8.743 ug/L (3.69%)



======= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Corn Oil spike 100ppb Zn.dth
Sample ID : Corn Oil spike 100ppb Zn
Creator determ.: Date : 2010-01-02 Time: 13:50:33
Modified by : Date : 2010-01-20 Time: 16:20:29

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl continous from previous Cd pb)
Remark2 : Deposition time 10s, total volume 10.65. stiring 1000rpm

---

Sample amount : 0.500 mL
Cell volume : 10.650 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 1.000ml

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-1.101	1.68	1.62	0.083	0.00	
1 - 2	-1.101	1.56				
2 - 1	-1.101	21.19	19.14	---	17.88	
2 - 2	-1.101	19.14				
3 - 1	-1.101	35.74	35.37	0.521	16.23	
3 - 2	-1.101	35.00				
4 - 1	-1.101	50.74	50.57	0.242	15.20	
4 - 2	-1.101	50.40				

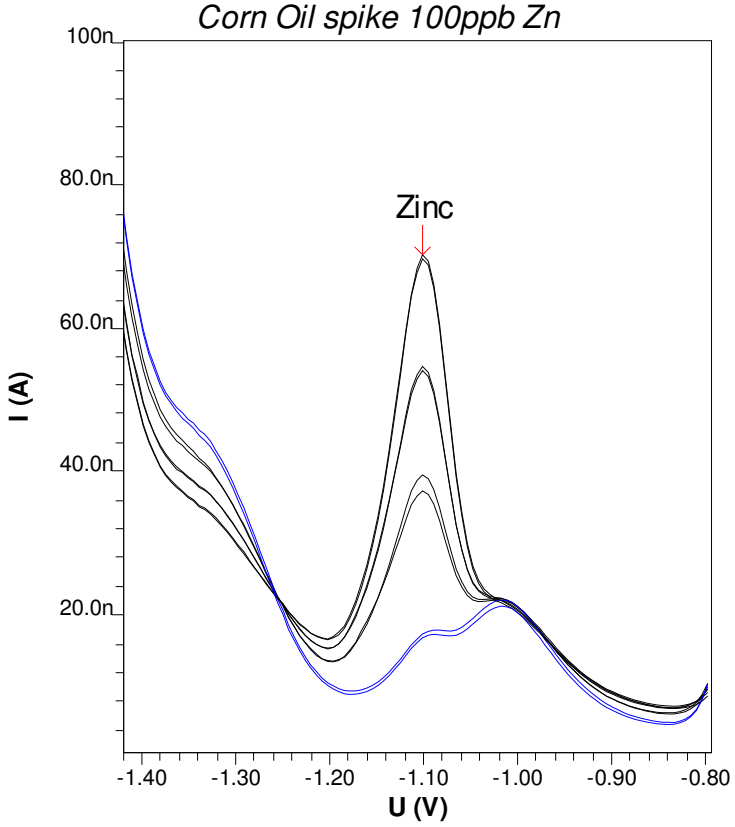
Solutions

No.	Content	Predose (mL)

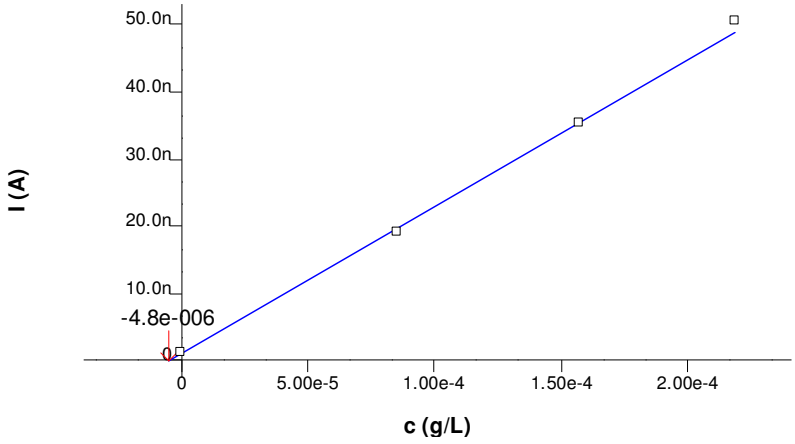
Final results		+/- Res. dev.	%	Comments
Zinc:				
default	= 101.450 ug/L	30.967	30.524	

Determination of Zn in edible Oil

Corn Oil spike 100ppb Zn



Zinc
c = 101.450 ug/L
+/- 30.967 ug/L (30.52%)



======== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Cooking Oil Cd Pb.dth
Sample ID : Palm Cooking Oil Cd Pb
Creator determ.: Date : 2010-01-02 Time: 12:54:28
Modified by : Date : 2010-01-21 Time: 21:59:26

Method : Determination of Cd and Pb in edible Oil with Acetate buffer
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.10 smpl + 10ml UPW)+0.25ml Acetate buffer
Remark2 : Deposition time 30 s

Sample amount : 0.100 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.668	1.95	2.10	0.200	0.00	
1 - 2	-0.668	2.24				
2 - 1	-0.668	11.04	10.36	---	8.27	
2 - 2	-0.668	10.36				
3 - 1	-0.668	18.42	17.70	1.021	7.34	
3 - 2	-0.668	16.98				
4 - 1	-0.668	24.87	24.87	---	7.17	
4 - 2	-0.668	22.29				

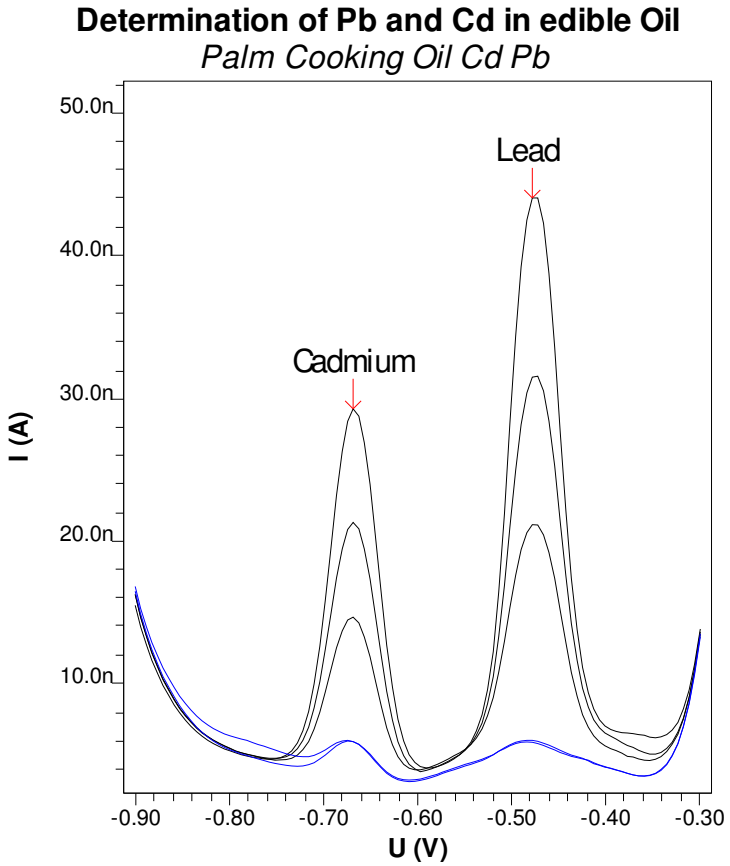
Substance : Lead
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.477	2.67	2.58	0.128	0.00	
1 - 2	-0.477	2.49				
2 - 1	-0.477	19.29	16.85	---	14.26	
2 - 2	-0.477	16.85				
3 - 1	-0.477	31.65	29.35	3.252	12.50	
3 - 2	-0.471	27.05				
4 - 1	-0.477	39.01	39.01	---	9.66	
4 - 2	-0.471	33.52				

Solutions

No.	Content	Predose (mL)
1	Standard For Cd and Pb	-------

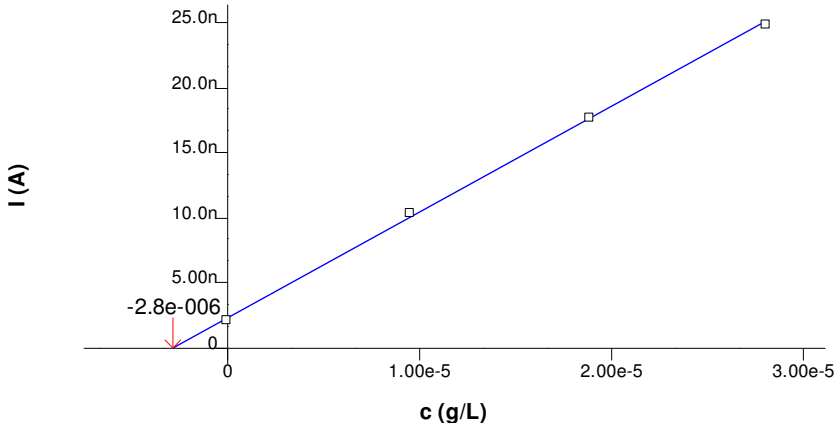
Final results			+/-	Res. dev.	%	Comments
Cadmium:						
default	=	58.409 ug/L		8.421	14.417	
Lead :						
default	=	46.722 ug/L		12.086	25.868	



Cadmium

c = 58.409 ug/L

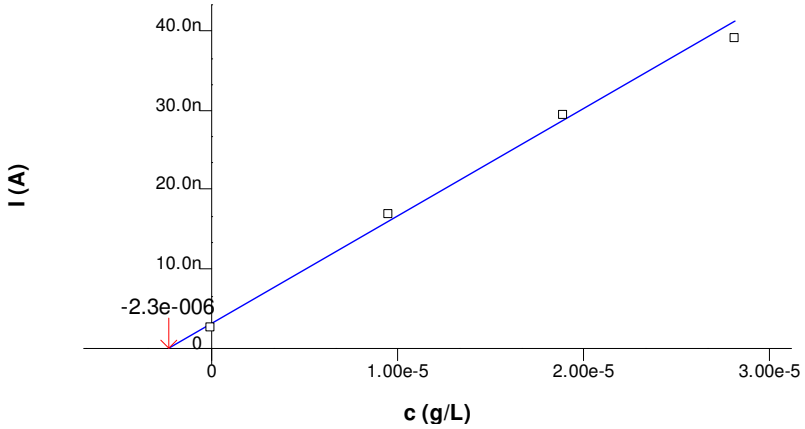
+/- 8.421 ug/L (14.42%)



Lead

c = 46.722 ug/L

+/- 12.086 ug/L (25.87%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Cooking Oil Ni.dth
Sample ID : Palm Cooking Oil Ni
Creator determ.: Date : 2010-01-02 Time: 12:25:06
Modified by : Date : 2010-01-21 Time: 22:29:40

Method : Ni Determination in Edible Oil (continous method).mth
Title : Ni Determination in Edible Oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : deposition time 5s, total vol 14.75

Sample amount : 0.500 mL
Cell volume : 14.750 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.959	-38.5	-38.6	0.087	0.0	
1 - 2	-0.959	-38.6				
2 - 1	-0.970	-75.7	-75.7	0.030	-37.1	
2 - 2	-0.970	-75.7				
3 - 1	-0.970	-107.6	-108.1	0.675	-32.4	
3 - 2	-0.970	-108.5				
4 - 1	-0.970	-145.2	-142.4	3.936	-34.3	
4 - 2	-0.970	-139.6				

Solutions

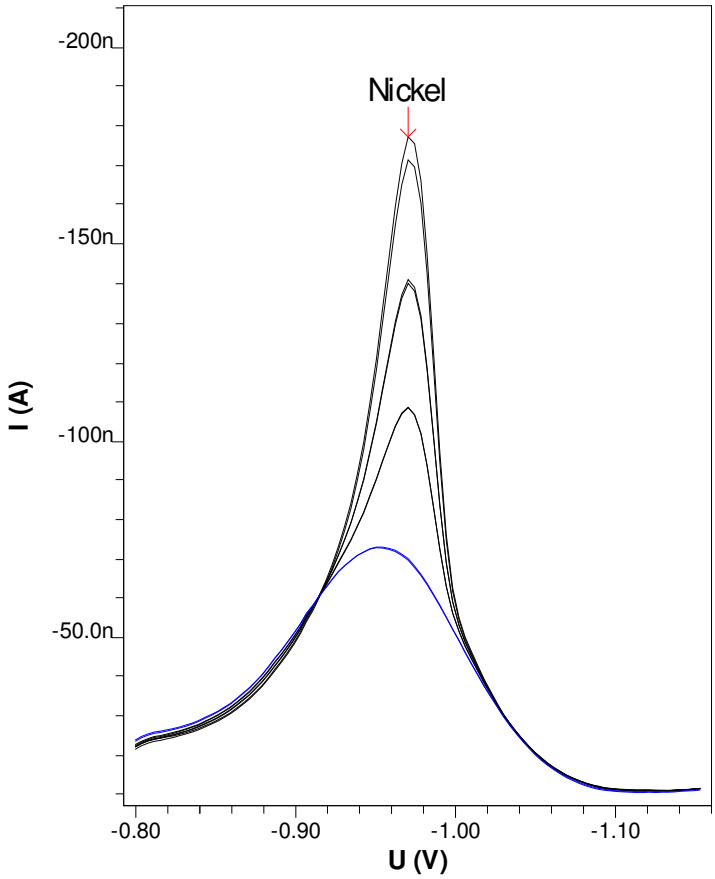
No.	Content	Predose (mL)

Final results	+/-	Res. dev.	%	Comments

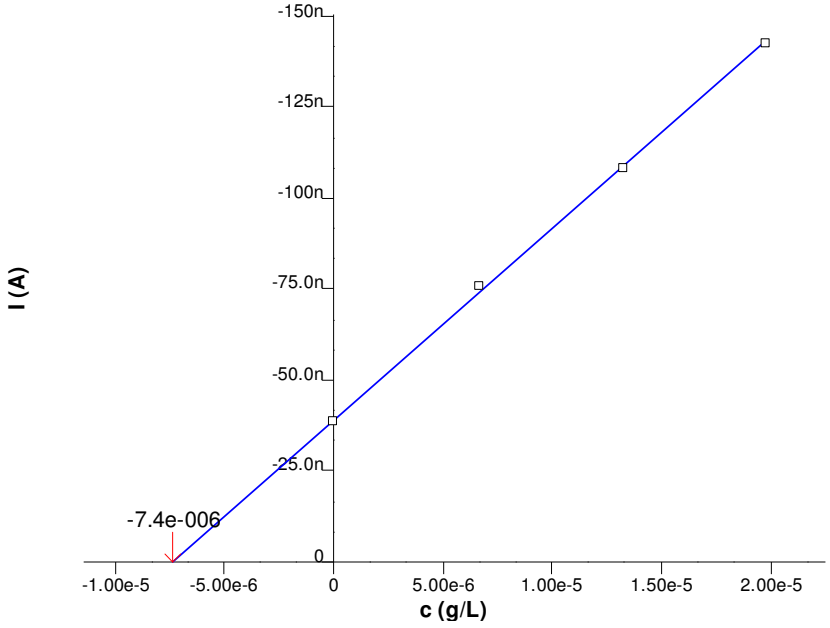
Nickel:
default = 216.935 ug/L 5.318 2.452

Ni Determination in Edible Oil

Palm Cooking Oil Ni



Nickel
c = 216.935 ug/L
+/- 5.318 ug/L (2.45%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Cooking Oil Zn.dth
Sample ID : Palm Cooking Oil Zn
Creator determ.: Date : 2010-01-02 Time: 12:35:44
Modified by : Date : 2010-01-21 Time: 22:13:34

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50smpl continous from previous Cd pb)
Remark2 : Deposition time 40s, total volume 10.65. stiring 1000rpm

---

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : Variable. Add 1 - 0.500ml, Add 2 – 0.500ml, Add 3-1.000ml

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-1.106	4.10	4.10	0.004	0.00	
1 - 2	-1.100	4.11				
2 - 1	-1.106	14.90	15.43	0.760	11.33	
2 - 2	-1.100	15.97				
3 - 1	-1.106	25.28	25.26	0.033	9.82	
3 - 2	-1.106	25.23				
4 - 1	-1.100	41.41	41.56	0.210	16.30	
4 - 2	-1.100	41.71				

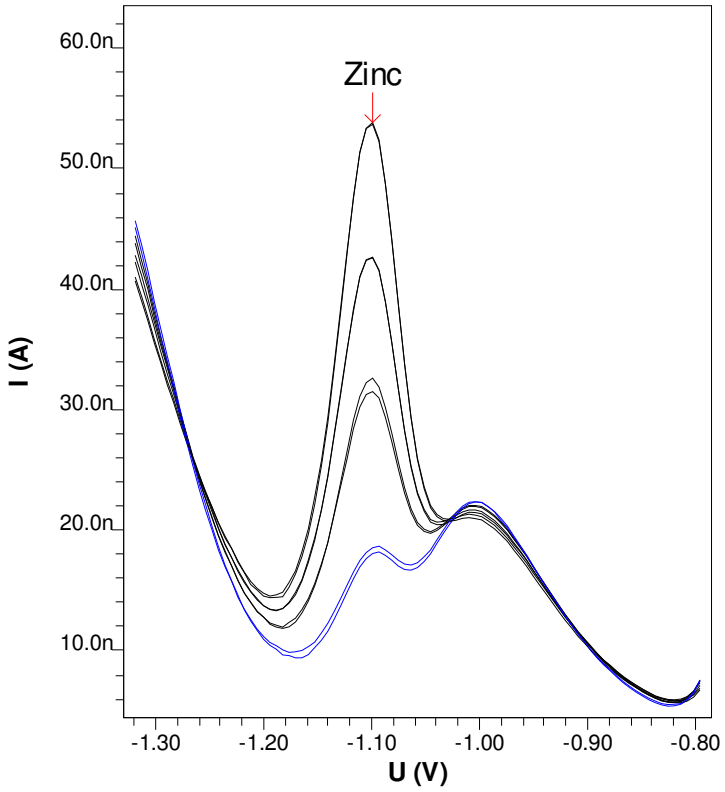
Solutions

No.	Content	Predose (mL)

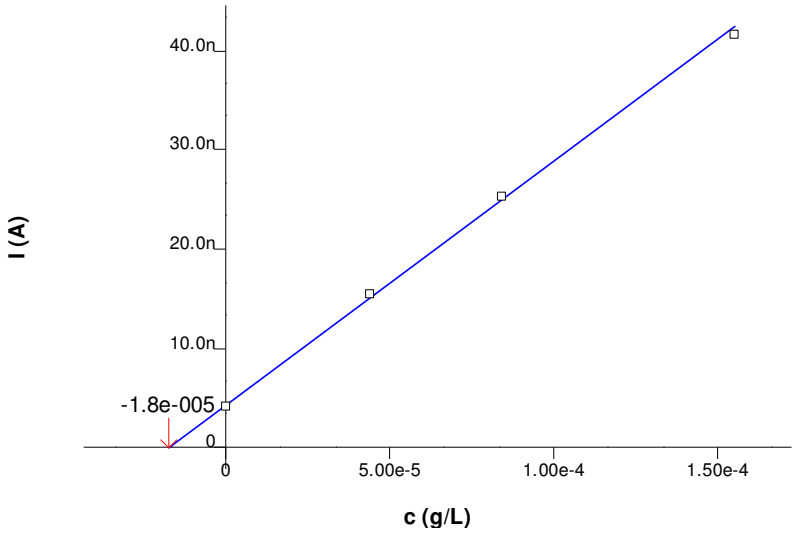
Final results	+/-	Res. dev.	%	Comments
Zinc:				
default = 375.716 ug/L		29.940	7.969	

Determination of Zn in edible Oil

Palm Cooking Oil Zn



Zinc
c = 375.716 ug/L
+/- 29.940 ug/L (7.97%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Olive oil Cd Pb dth
Sample ID : Olive oil Cd Pb
Creator determ.: Date : 2010-01-11 Time: 10:30:51
Modified by : Date : 2010-01-20 Time: 23:34:32

Method : Determination of Cd and Pb in edible Oil with Acetate buffer
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.5 smpl + 10ml UPW)+0.25ml Acetate Buffer
Remark2 : deposition time 50s

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.602	38.82	38.82	---	0.00	
1 - 2	-0.602	42.44				
2 - 1	-0.602	52.62	55.49	4.051	16.67	
2 - 2	-0.602	58.35				
3 - 1	-0.602	69.79	71.88	2.956	16.39	
3 - 2	-0.602	73.97				
4 - 1	-0.602	85.70	89.18	---	17.31	
4 - 2	-0.602	89.18				

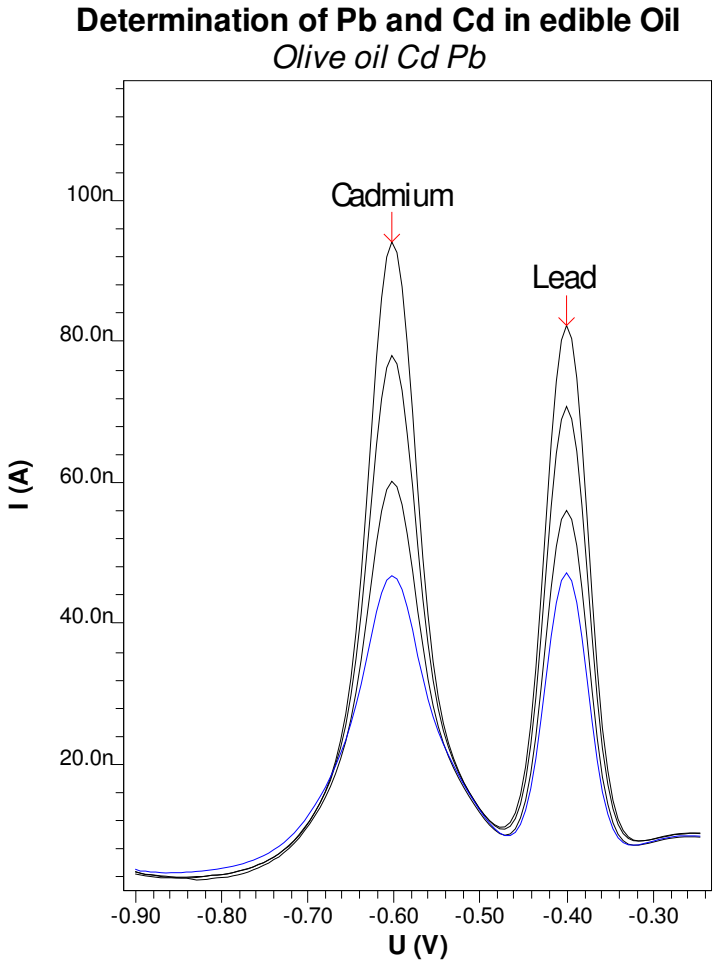
Substance : Lead
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.400	37.90	37.90	---	0.00	
1 - 2	-0.400	39.99				
2 - 1	-0.400	46.74	49.21	3.492	11.31	
2 - 2	-0.400	51.68				
3 - 1	-0.400	60.77	62.00	1.738	12.79	
3 - 2	-0.400	63.23				
4 - 1	-0.400	72.13	73.30	---	11.30	
4 - 2	-0.400	73.30				

Solutions

No.	Content	Predose (mL)
1	Standard For Cd and Pb	-------

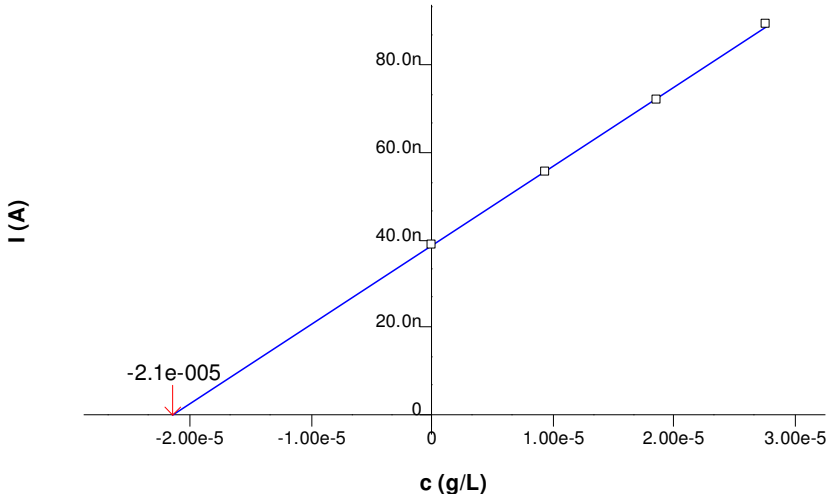
Final results			+/-	Res. dev.	%	Comments
Cadmium:						
default	=	443.044 ug/L		27.252	6.151	
Lead :						
default	=	601.537 ug/L		39.827	6.621	



Cadmium

c = 443.044 ug/L

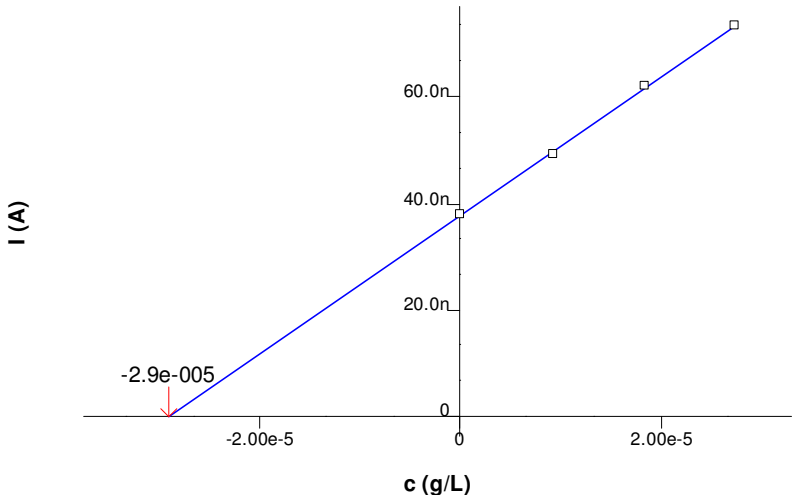
+/- 27.252 ug/L (6.15%)



Lead

c = 601.537 ug/L

+/- 39.827 ug/L (6.62%)



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Olive Oil Ni (C).dth
Sample ID : Olive Oil Ni
Creator determ.: Date : 2010-01-11 Time: 11:10:44
Modified by : Date : 2010-01-21 Time: 16:15:18

Method : Ni Determination in Edible Oil (continous method).mth
Title : Ni Determination in Edible Oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : deposition time 5s, Total Volume 14.750ml

Sample amount : 0.500 mL
Cell volume : 14.750 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.963	-48.2	-48.2	0.104	0.0	
1 - 2	-0.963	-48.3				
2 - 1	-0.966	-85.7	-86.6	1.281	-38.4	
2 - 2	-0.966	-87.5				
3 - 1	-0.970	-126.5	-126.4	0.215	-39.7	
3 - 2	-0.970	-126.2				
4 - 1	-0.970	-164.6	-164.3	0.551	-37.9	
4 - 2	-0.970	-163.9				

Solutions

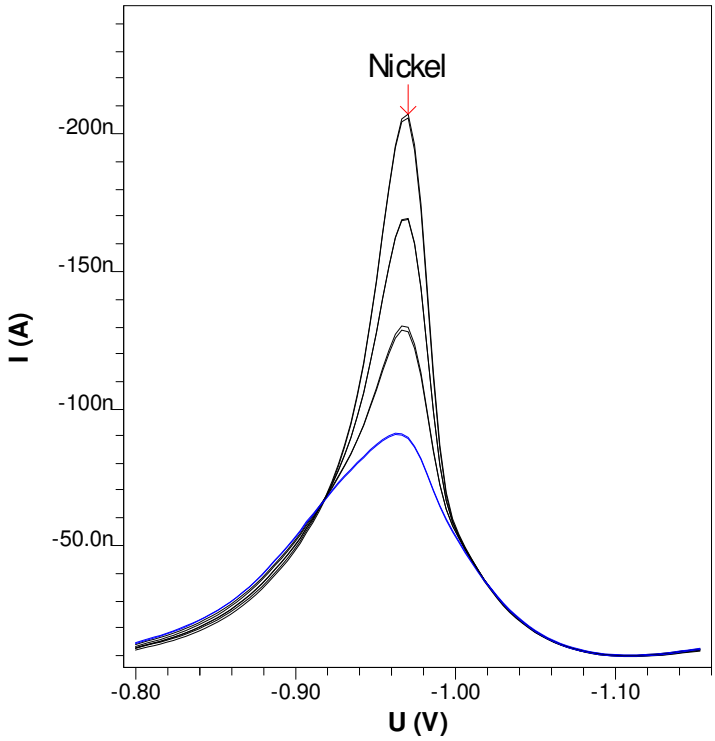
No.	Content	Predose (mL)

Final results	+/-	Res. dev.	%	Comments

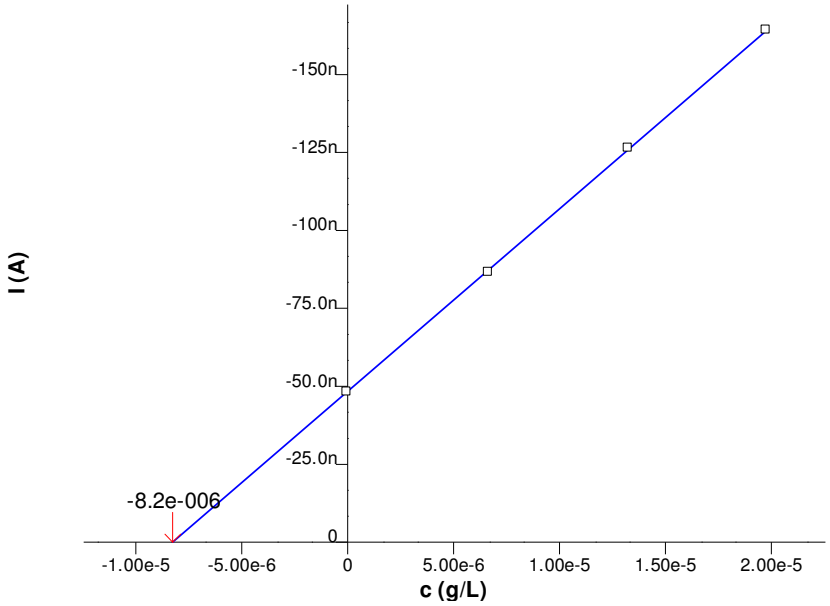
Nickel:
default = 242.216 ug/L 2.750 1.136

Ni Determination in Edible Oil

Olive Oil Ni



Nickel
c = 242.216 ug/L
+/- 2.750 ug/L (1.14%)



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Olive Oil Zn (C).dth
Sample ID : Olive Oil Zn
Creator determ.: Date : 2010-01-11 Time: 10:51:07
Modified by : Date : 2010-01-21 Time: 16:46:10

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl continous from previous Cd pb)
Remark2 : Deposition time 90s, total volume 10.65. stiring 1000rpm

---

Sample amount : 0.500 mL
Cell volume : 10.650 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 250.00 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.999	12.60	12.55	0.083	0.00	
1 - 2	-1.004	12.49				
2 - 1	-0.999	34.44	34.37	0.097	21.83	
2 - 2	-0.999	34.30				
3 - 1	-0.999	54.78	53.34	2.042	18.97	
3 - 2	-0.999	51.89				
4 - 1	-0.999	72.41	73.27	1.211	19.93	
4 - 2	-0.999	74.12				

Solutions

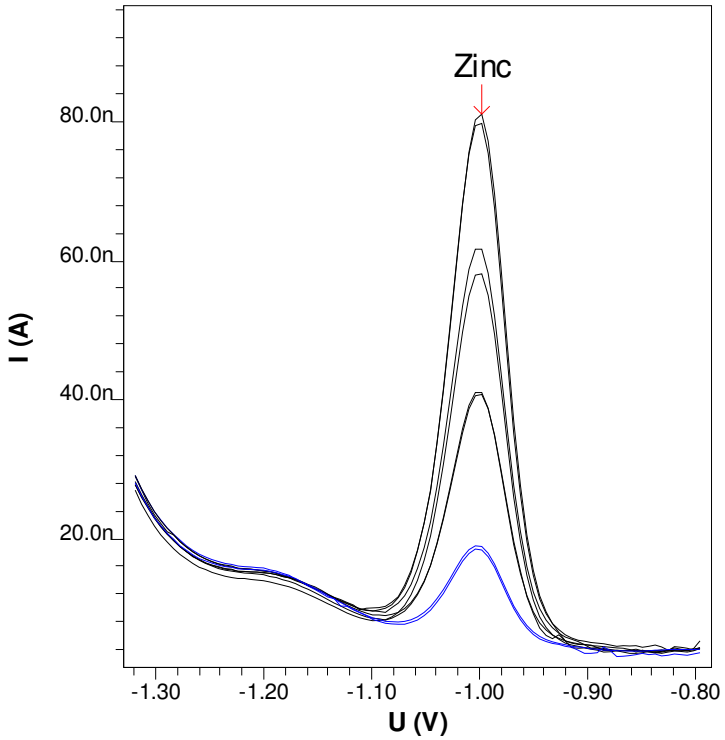
No.	Content	Predose (mL)

Final results	+/-	Res. dev.	%	Comments

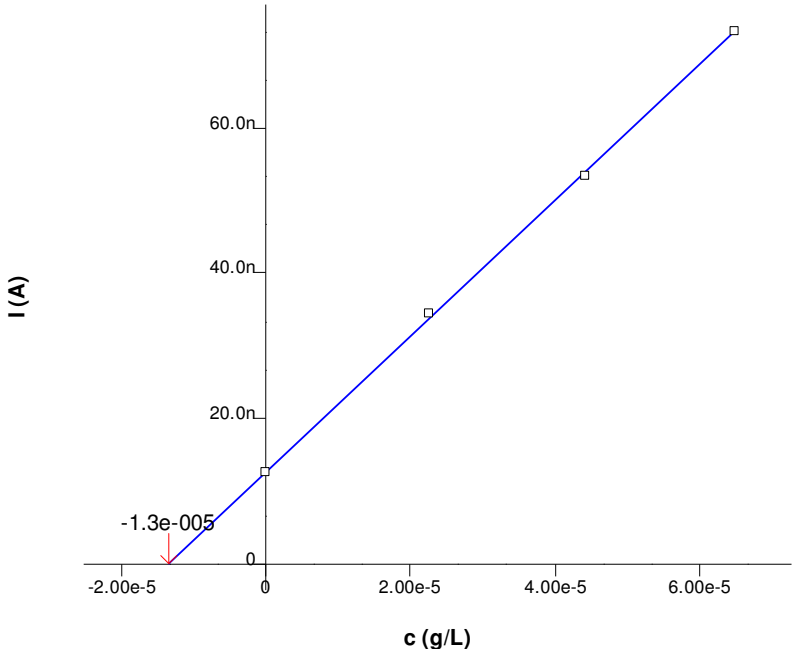
Zinc:
default = 286.799 ug/L 8.017 2.795

Determination of Zn in edible Oil

Olive Oil Zn



Zinc
c = 286.799 ug/L
+/- 8.017 ug/L (2.80%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Olive oil Cd Pb Spike 100ppb.dth
Sample ID : Olive oil Spike 100ppb Cd Pb
Creator determ.: Date : 2010-01-12 Time: 16:32:12
Modified by : --- Date : 2010-01-20 Time: 23:40:43

Method : Determination of Cd and Pb in edible Oil with Acetate Buffer.mth
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.50 smpl + 10ml UPW)+0.25ml Acetate Buffer
Remark2 : Deposition time 40s

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. Std : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.602	48.0	50.9	---	0.0	
1 - 2	-0.602	50.9				
2 - 1	-0.602	62.2	65.9	5.295	15.0	
2 - 2	-0.602	69.7				
3 - 1	-0.602	79.2	83.2	5.609	17.2	
3 - 2	-0.602	87.1				
4 - 1	-0.602	99.2	105.6	---	22.4	
4 - 2	-0.602	105.6				

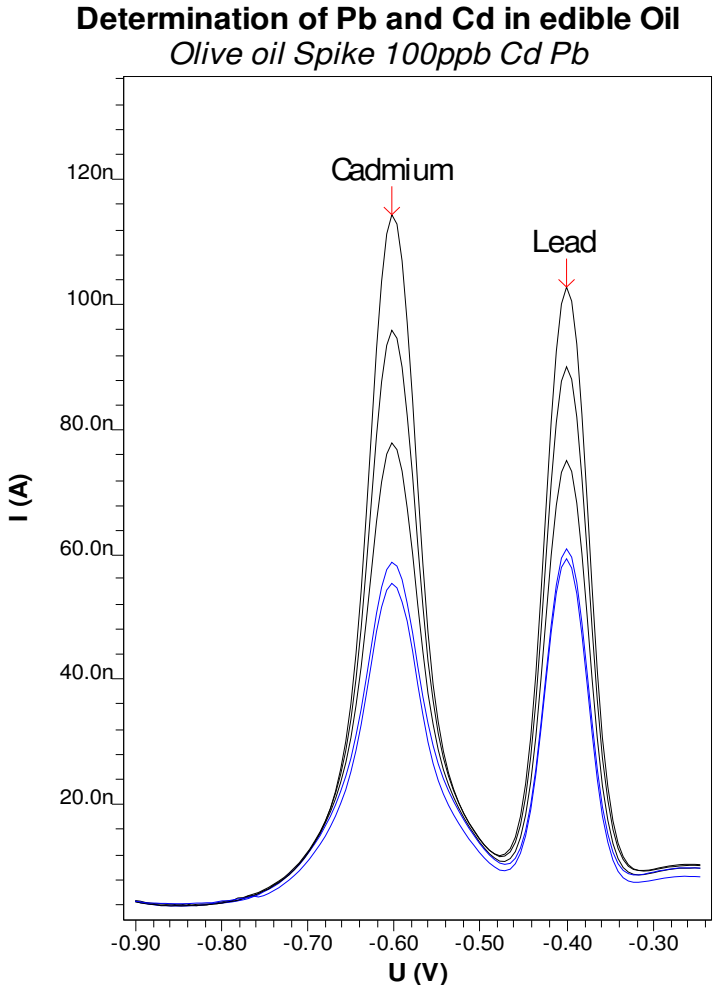
Substance : Lead
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.400	50.88	51.30	---	0.00	
1 - 2	-0.400	51.30				
2 - 1	-0.400	58.88	62.04	4.469	10.74	
2 - 2	-0.400	65.20				
3 - 1	-0.400	72.91	76.19	4.647	14.15	
3 - 2	-0.400	79.48				
4 - 1	-0.400	85.59	92.20	---	16.01	
4 - 2	-0.400	92.20				

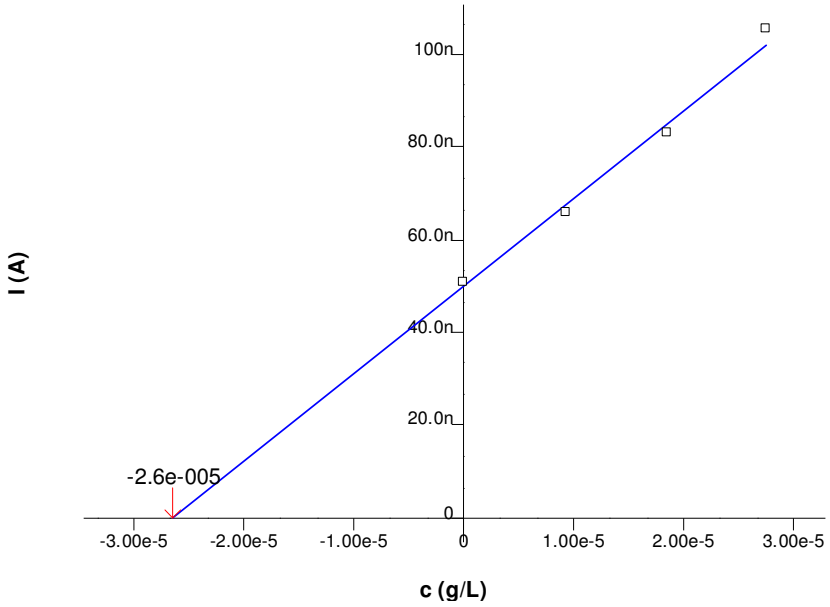
Solutions

No.	Content	Predose (mL)
1	Standard For Cd and Pb	-------

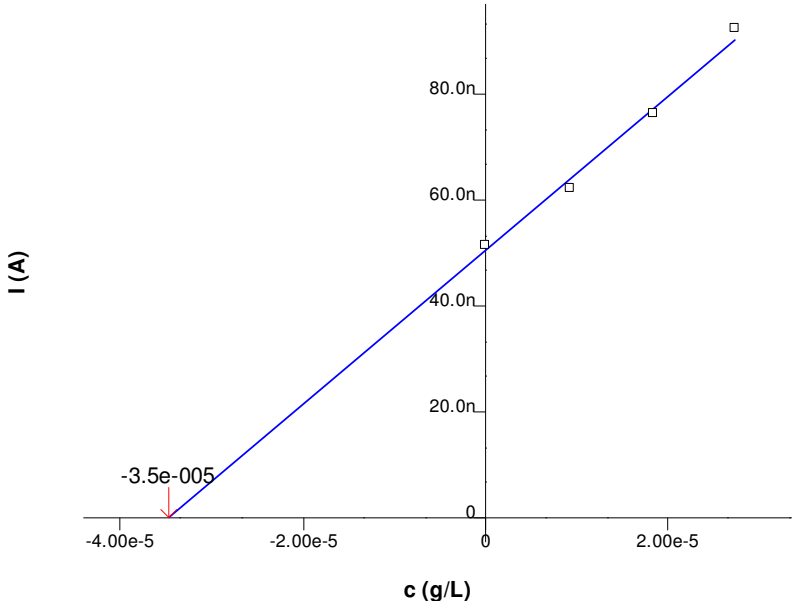
Final results		+/-	Res. dev.	%	Comments
Cadmium:					
default	= 544.882 ug/L		56.415	10.354	
Lead :					
default	= 714.902 ug/L		74.995	10.490	



Cadmium
c = 544.882 ug/L
+/- 56.415 ug/L (10.35%)



Lead
c = 714.902 ug/L
+/- 74.995 ug/L (10.49%)



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Olive Oil spike 100ppb Ni dth
Sample ID : Olive Oil spike 100ppb Ni
Creator determ.: Date : 2010-01-12 Time: 17:15:02
Modified by : Date : 2010-01-21 Time: 16:32:08

Method : Ni Determination in Edible Oil (continous method).mth
Title : Ni Determination in Edible Oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : deposition 5s, Total Volume 14.75

Sample amount : 0.500 mL
Cell volume : 14.750 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.966	-63.3	-70.0	9.472	0.0	
1 - 2	-0.966	-76.7				
2 - 1	-0.966	-102.4	-110.5	11.446	-40.5	
2 - 2	-0.966	-118.6				
3 - 1	-0.966	-137.8	-151.6	19.550	-41.1	
3 - 2	-0.966	-165.4				
4 - 1	-0.966	-171.7	-184.1	17.563	-32.5	
4 - 2	-0.966	-196.5				

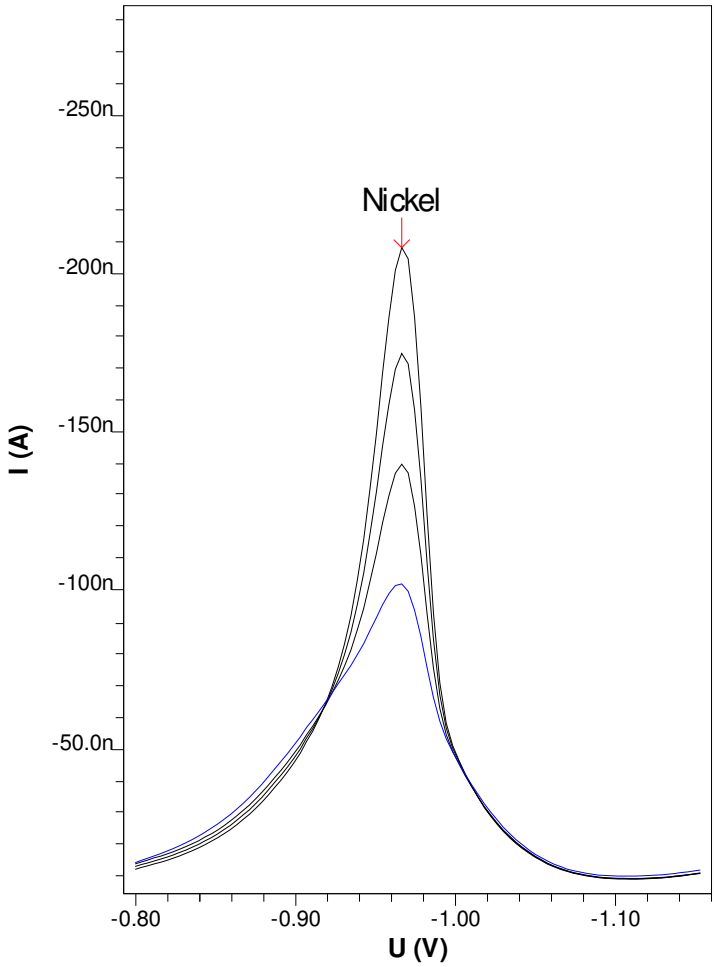
Solutions

No.	Content	Predose (mL)

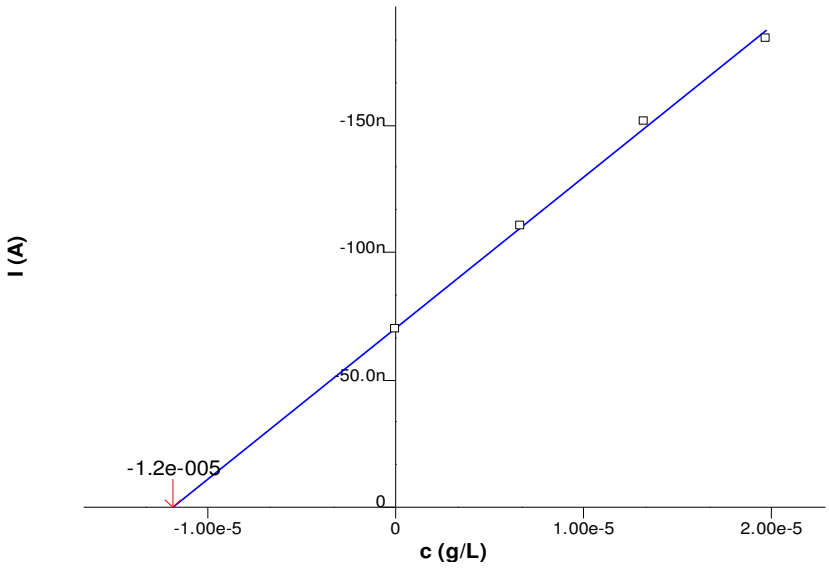
Final results	+/- Res. dev.	%	Comments
Nickel:			
default = 349.268 ug/L	53.782	15.398	

Ni Determination in Edible Oil

Olive Oil spike 100ppb Ni



Nickel
c = 349.268 ug/L
+/- 53.782 ug/L (15.40%)



======== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Olive oil Spike 100ppb Zn.dth
Sample ID : Olive oil Spike 100ppb Zn
Creator determ. : Date : 2010-01-12 Time: 16:53:14
Modified by : Date : 2010-01-21 Time: 16:59:48

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl continous from previous Cd pb)
Remark2 : Deposition time 90s, total volume 10.65.

Sample amount : 0.500 mL
Cell volume : 10.650 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.999	15.76	15.76	---	0.00	
1 - 2	-0.999	16.42				
2 - 1	-0.999	35.26	36.03	1.095	20.27	
2 - 2	-0.999	36.80				
3 - 1	-0.999	54.22	54.42	0.276	18.39	
3 - 2	-0.999	54.61				
4 - 1	-0.999	71.79	71.82	0.043	17.41	
4 - 2	-0.999	71.85				

Solutions

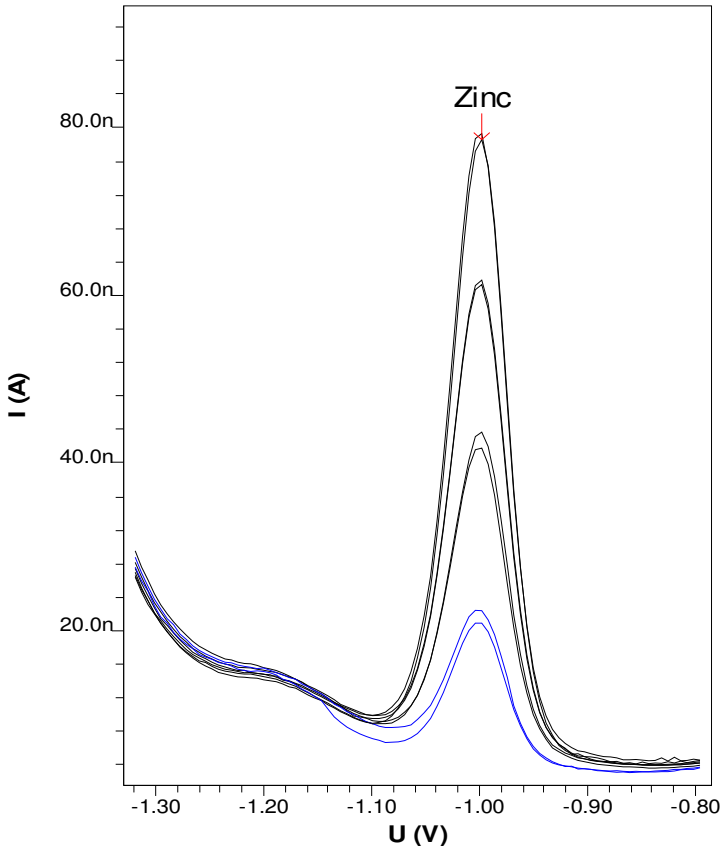
No.	Content	Predose (mL)

Final results	+/-	Res. dev.	%	Comments

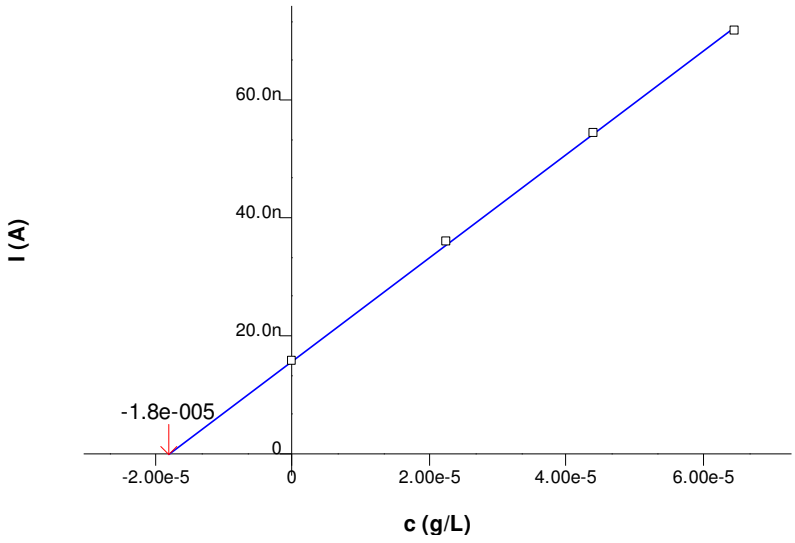
Zinc:
default = 384.359 ug/L 7.390 1.923

Determination of Zn in edible Oil

Olive oil Spike 100ppb Zn



Zinc
c = 384.359 ug/L
+/- 7.390 ug/L (1.92%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Oil Maj (Cd Pb).dth
Sample ID : Palm Oil Maj
Creator determ.: Date : 2009-12-24 Time: 9:35:05
Modified by : --- Date : 2010-01-22 Time: 12:00.03

Method : Determination of Cd and Pb in edible Oil with Acetate buffer
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.5 smpl + 10ml UPW)+0.25ml acetate buffer
Remark2 : deposition time 30s

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.696	8.21	8.27	0.096	0.00	
1 - 2	-0.702	8.34				
1 - 3	---	---				
2 - 2	---	---	19.82	0.237	11.54	
2 - 2	-0.655	19.99				
2 - 3	-0.661	19.65				
3 - 1	-0.661	32.02	30.44	1.412	10.63	
3 - 2	-0.655	29.29				
3 - 3	-0.655	30.03				

Substance : Lead
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.458	2.49	2.46	0.045	0.00	
1 - 2	-0.458	2.42				
1 - 3	-0.464	2.71				
2 - 1	-0.470	8.65	8.04	0.529	5.58	
2 - 2	-0.452	7.74				
2 - 3	-0.452	7.73				
3 - 1	-0.446	15.44	14.88	0.583	6.84	

3 - 2 -0.446 14.28
3 - 3 -0.446 14.92

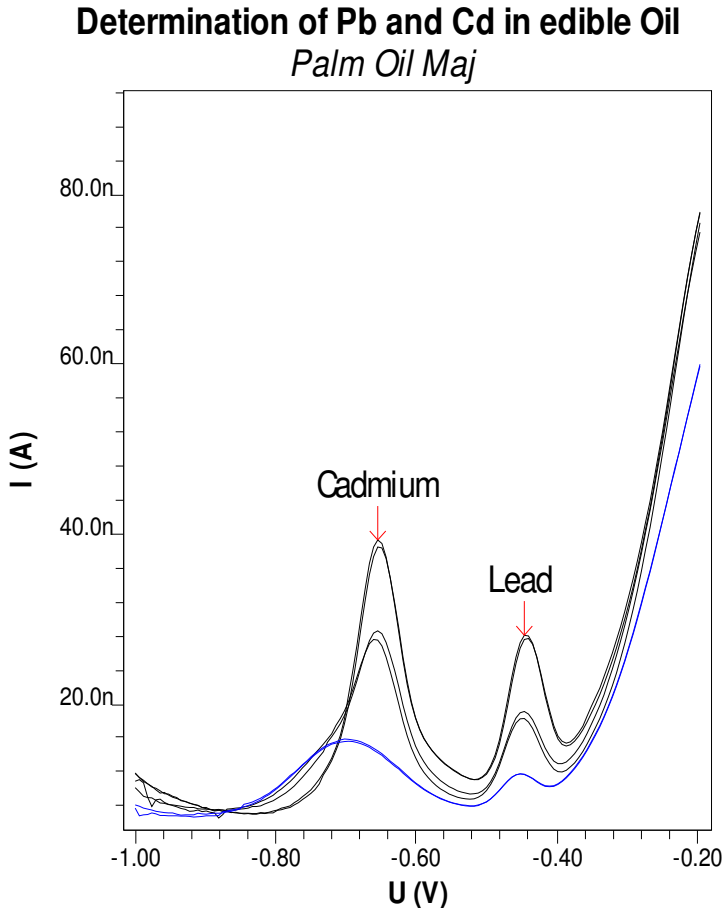
Solutions

No.	Content	Predose (mL)

Final results	+/- Res. dev.	%	Comments

Cadmium:
default = 145.251 ug/L 5.130 3.532

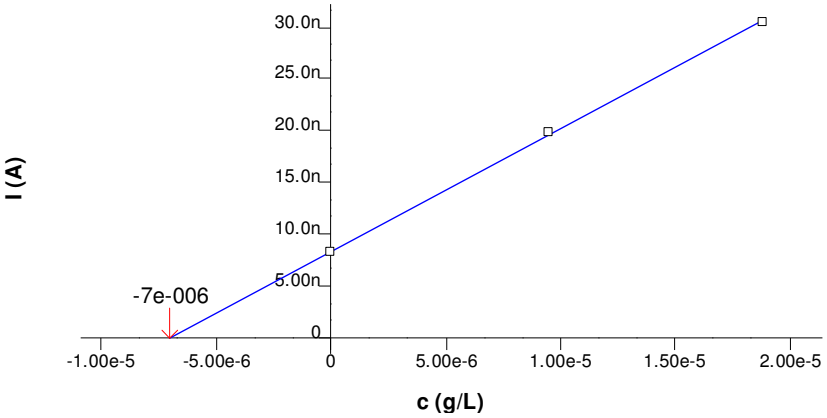
Lead :
default = 70.035 ug/L 10.421 14.880



Cadmium

c = 145.251 ug/L

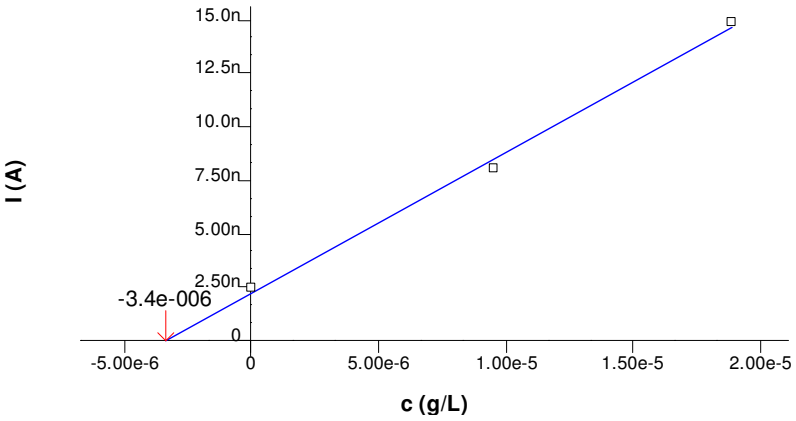
+/- 5.130 ug/L (3.53%)



Lead

c = 70.035 ug/L

+/- 10.421 ug/L (14.88%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Oil Maj Ni.dth
Sample ID : Palm Oil Maj Ni
Creator determ.: Date : 2009-12-24 Time: 9:27:29
Modified by : Date : 2010-01-22 Time: 14:39:27

Method : Ni Determination in Edible Oil (continous method).mth
Title : Ni Determination in Edible Oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : deposition time 5s, Total Volume 11.400ml

Sample amount : 0.500 mL
Cell volume : 11.4000 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 50.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.994	-16.39	-16.48	0.110	0.00	
1 - 2	-0.994	-16.61				
1 - 3	-0.994	-16.44				
2 - 1	-0.994	-32.56	-33.20	0.773	-16.72	
2 - 2	-0.994	-32.97				
2 - 3	-0.994	-34.06				
3 - 1	-0.994	-49.28	-47.49	1.601	-14.29	
3 - 2	-0.994	-46.20				
3 - 3	-0.994	-46.99				

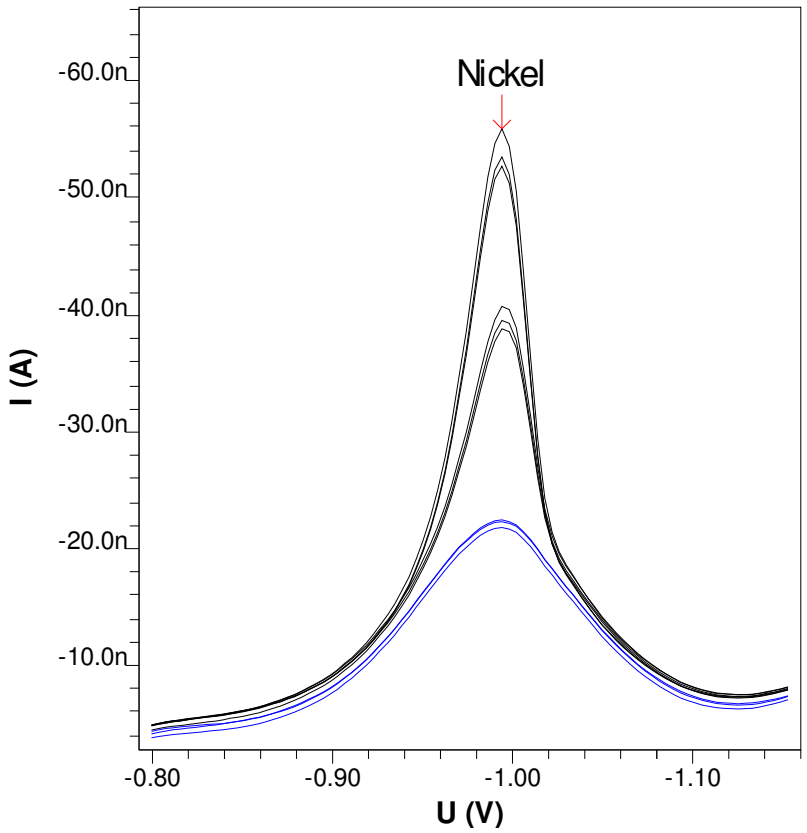
Solutions

No.	Content	Predose (mL)

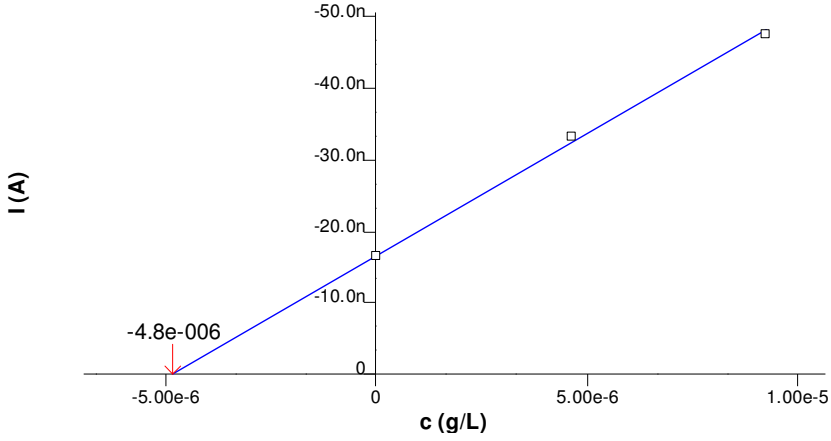
Final results	+/-	Res. dev.	%	Comments
Nickel:				
default = 103.199 ug/L		3.126		3.029

Ni Determination in Edible Oil

Palm Oil Ni



Nickel
c = 103.199 ug/L
+/- 3.126 ug/L (3.03%)



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : MC Palm Oil Maj Zn .dth
Sample ID : MC Palm Oil Maj Zn
Creator determ.: Date : 2009-12-24 Time: 9:55:01
Modified by : Date : 2010-01-22 Time: 14:21:21

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl continous from previous Cd pb)
Remark2 : Deposition time 90s, total volume 10.65. stiring 1000rpm

---

Sample amount : 0.500 mL
Cell volume : 10.650 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 250.00 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.999	20.49	20.50	0.019	0.00	
1 - 2	-0.999	20.52				
2 - 1	-0.999	44.60	44.59	0.011	24.09	
2 - 2	-0.999	44.59				
3 - 1	-0.999	65.69	65.51	0.257	20.91	
3 - 2	-0.999	65.33				
4 - 1	-0.999	89.10	87.29	2.557	21.78	
4 - 2	-0.999	85.48				

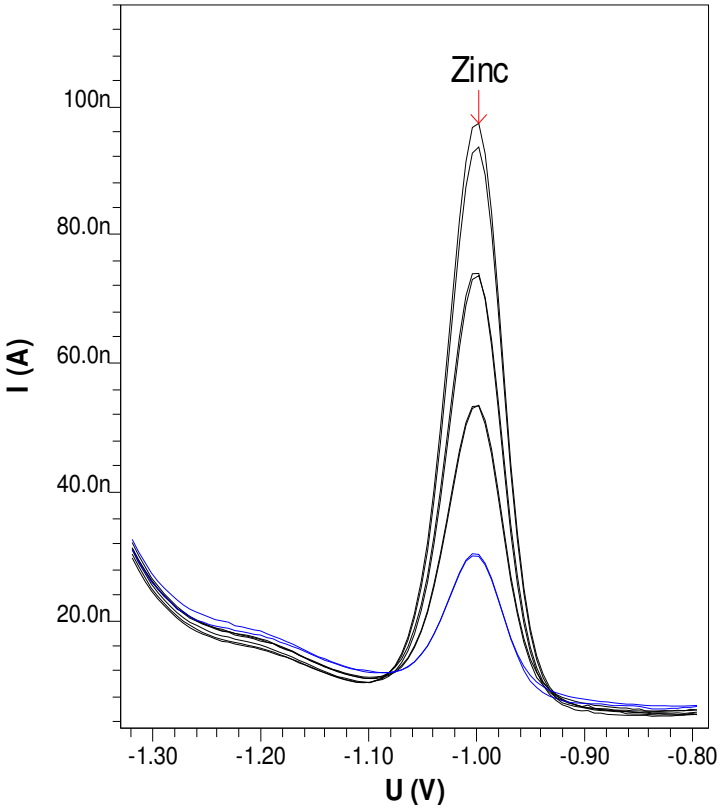
Solutions

No.	Content	Predose (mL)

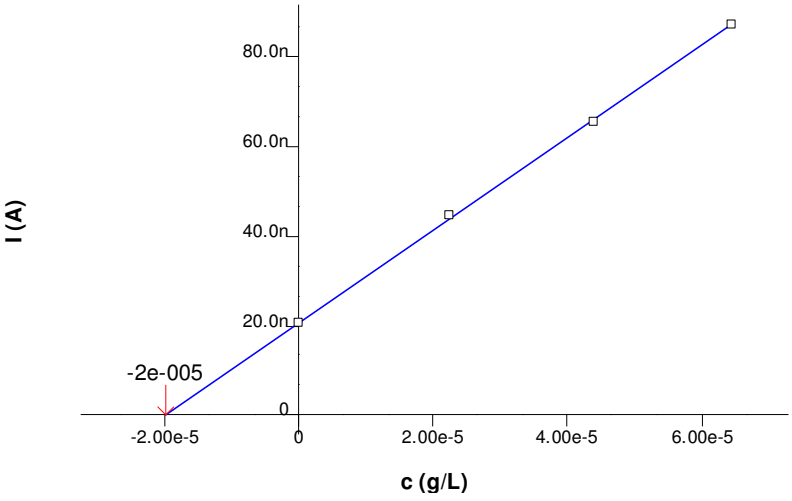
Final results		+/- Res. dev.	%	Comments
Zinc:				
default	= 422.711 ug/L	8.712	2.061	

Determination of Zn in edible Oil

Palm Oil Maj Zn



Zinc
c = 422.711 ug/L
+/- 8.712 ug/L (2.06%)



======= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Oil Maj (Cd Pb).dth
Sample ID : Palm oil Maj (Cd Pb)
Creator determ.: Date : 2009-12-28 Time: 12:41:05
Modified by : Date : 2010-01-20 Time: 16:59:59

Method : Determination of Cd and Pb in edible Oil with Acetate buffer
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.5 smpl + 10ml UPW)+0.25ml acetate buffer
Remark2 : deposition time 30s

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.668	0.05	0.05	0.001	0.00	
1 - 2	-0.674	0.05				
2 - 1	-0.662	9.66	9.08	---	9.03	
2 - 2	-0.662	9.08				
3 - 1	-0.662	16.96	16.49	0.653	7.41	
3 - 2	-0.662	16.03				
4 - 1	-0.662	23.86	23.86	---	7.36	
4 - 2	-0.662	21.83				

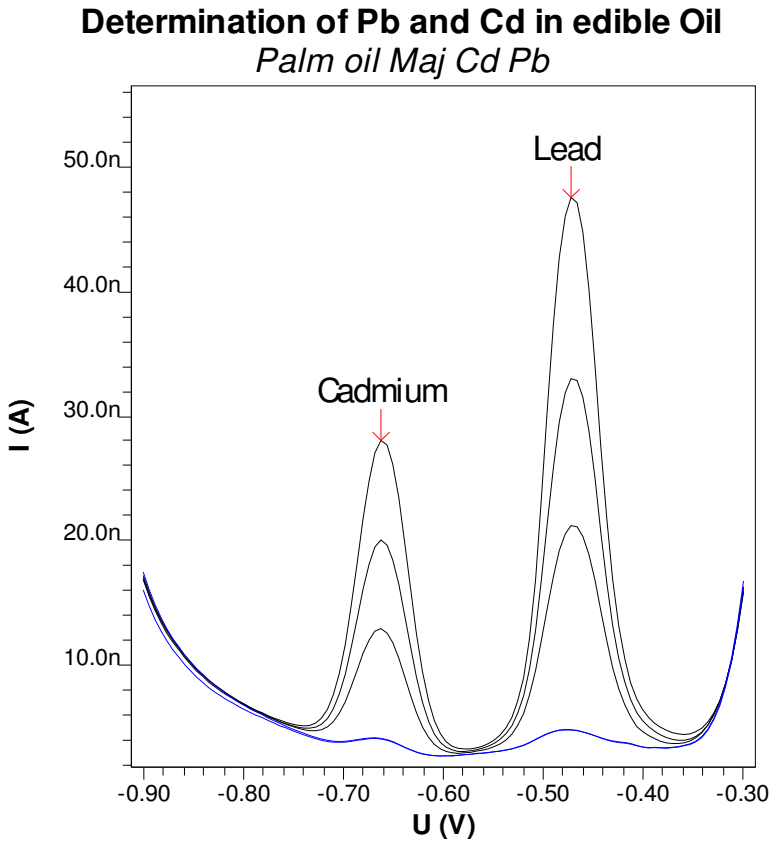
Substance : Lead
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.477	1.14	1.13	0.013	0.00	
1 - 2	-0.477	1.12				
2 - 1	-0.471	20.05	17.88	---	16.75	Not used
2 - 2	-0.471	17.88				
3 - 1	-0.471	33.07	31.28	2.526	13.40	
3 - 2	-0.471	29.50				
4 - 1	-0.471	43.75	43.75	---	12.47	
4 - 2	-0.471	38.35				

Solutions

No.	Content	Predose (mL)
1	Standard For Cd and Pb	-------

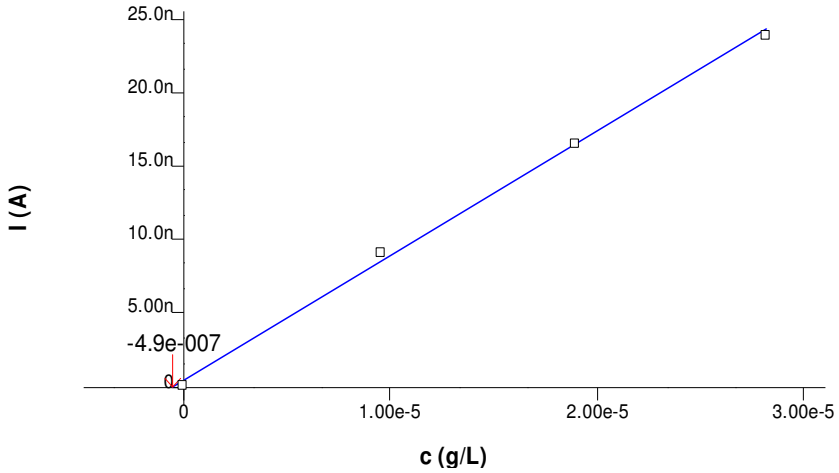
Final results			+/-	Res. dev.	%	Comments
Cadmium:						
default	=	10.160 ug/L		8.010	78.834	
Lead :						
default	=	21.967 ug/L		9.168	41.734	



Cadmium

c = 10.160 ug/L

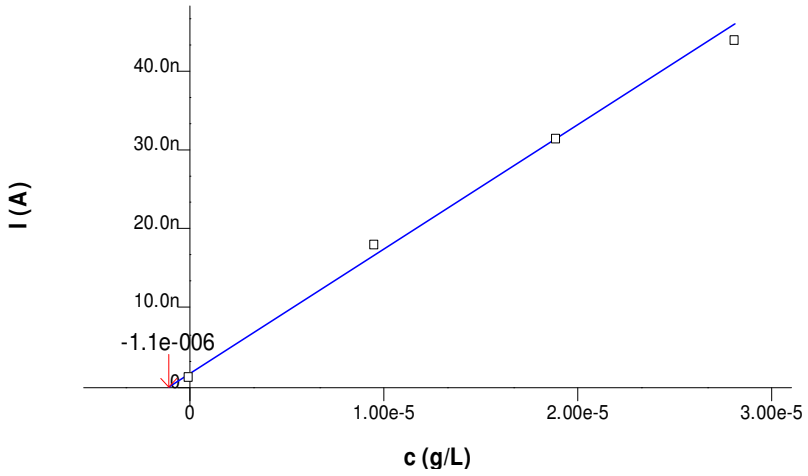
+/- 8.010 ug/L (78.83%)



Lead

c = 21.967 ug/L

+/- 9.168 ug/L (41.73%)



======== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Oil Maj Ni.dth
Sample ID : Palm Oil Maj Ni
Creator determ.: Date : 2009-12-28 Time: 13:20:30
Modified by : Date : 2010-01-20 Time: 17:38:47

Method : Ni Determination in Edible Oil (continous method).mth
Title : Ni Determination in Edible Oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : deposition time 5s, total volume 14.75

Sample amount : 0.500 mL
Cell volume : 14.750 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.963	-32.8	-32.9	0.165	0.0	
1 - 2	-0.963	-33.1				
2 - 1	-0.970	-70.3	-71.8	2.206	-38.9	
2 - 2	-0.970	-73.4				
3 - 1	-0.970	-110.5	-111.3	1.141	-39.4	
3 - 2	-0.970	-112.1				
4 - 1	-0.970	-151.0	-150.3	1.012	-39.0	
4 - 2	-0.970	-149.6				

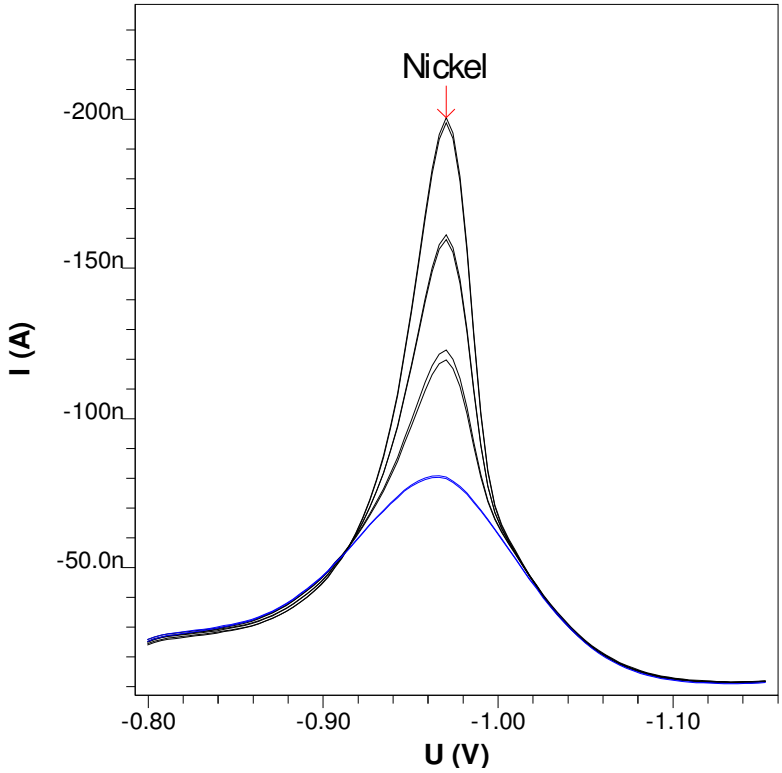
Solutions

No.	Content	Predose (mL)

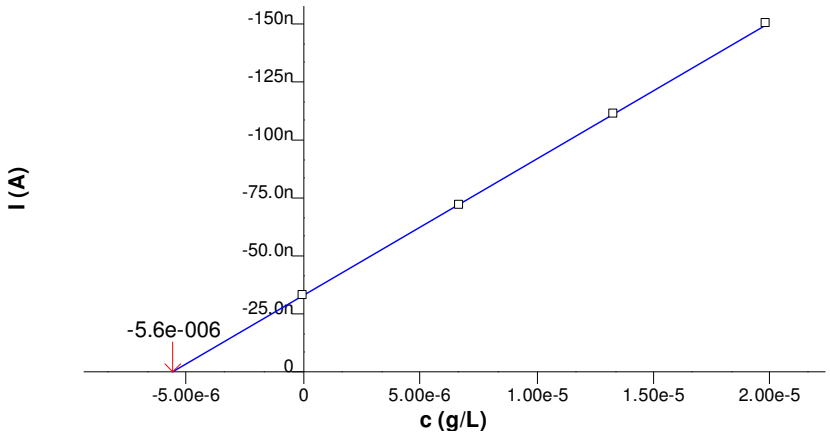
Final results	+/-	Res. dev.	%	Comments
Nickel:				
default = 164.607 ug/L	3.001		1.823	

Ni Determination in Edible Oil

Palm Oil Maj (bakery) Ni



Nickel
c = 164.607 ug/L
+/- 3.001 ug/L (1.82%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Oil Maj Zn.dth
Sample ID : Palm Oil Maj Zn
Creator determ.: Date : 2009-12-28 Time: 13:03:02
Modified by : Date : 2010-01-20 Time: 17:58:52

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl continous from previous Cd pb)
Remark2 : Deposition time 90s, total volume 10.65

Sample amount : 0.500 mL
Cell volume : 10.650 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.00 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-1.100	7.32	7.85		0.00	Not used
1 - 2	-1.100	7.85				
2 - 1	-1.100	16.91	17.17	0.370	9.32	
2 - 2	-1.100	17.43				
3 - 1	-1.100	24.63	24.67	0.058	7.50	
3 - 2	-1.100	24.71				
4 - 1	-1.100	34.74	33.89	1.204	9.22	
4 - 2	-1.100	33.04				

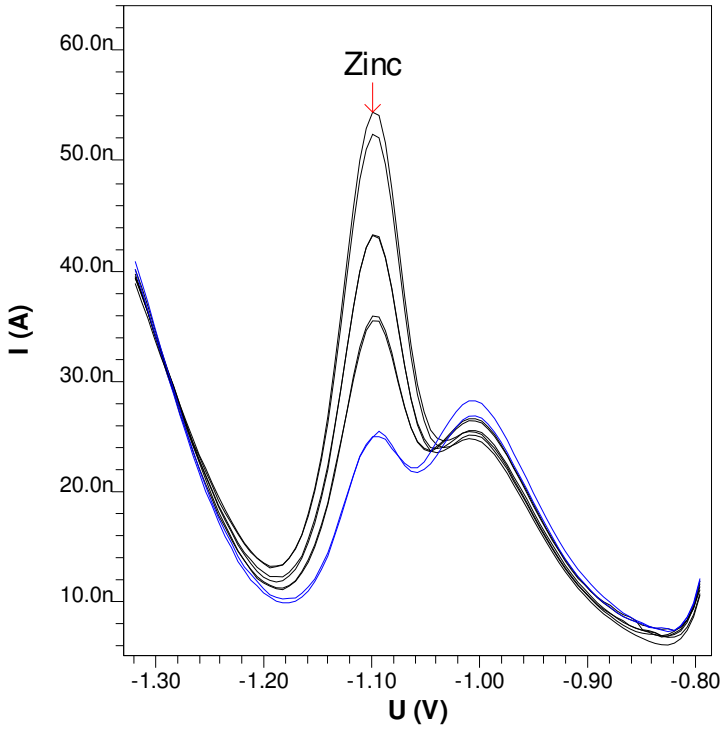
Solutions

No.	Content	Predose (mL)

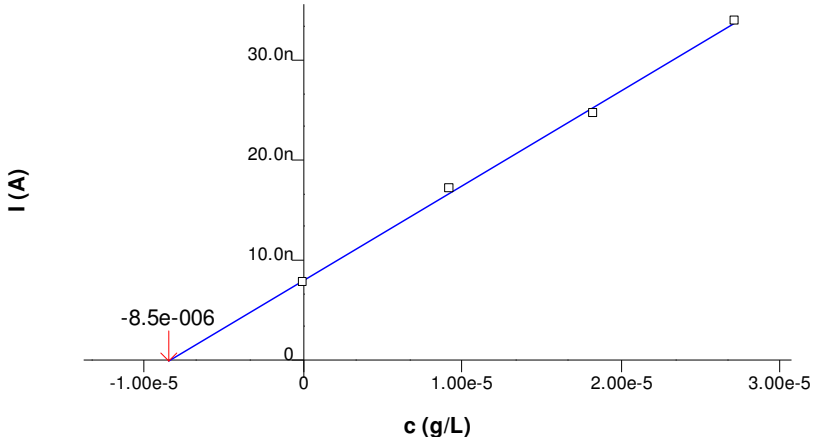
Final results	+/- Res. dev.	%	Comments
Zinc:			
default = 180.195 ug/L	10.126	5.619	

Determination of Zn in edible Oil

Palm oil Maj (bakery) Zn



Zinc
c = 180.195 ug/L
+/- 10.126 ug/L (5.62%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Oil Maj Spike 100 ppb Pb Cd.dth
Sample ID : Palm Oil Maj Spike 100 ppb Pb Cd
Creator determ.: Date : 2009-12-29 Time: 10.21:13
Modified by : Date : 2010-01-20 Time: 17:21:00

Method : Determination of Cd and Pb in edible Oil with NH4 buffer.mth
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.50 smpl + 10ml UPW)+0.25ml acetate buffer
Remark2 : Depostion time 30s

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. Std : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.662	3.23	3.23	---	0.00	
1 - 2	-0.662	3.00				
2 - 1	-0.662	11.85	11.43	0.601	8.20	
2 - 2	-0.662	11.01				
3 - 1	-0.662	19.21	18.32	1.254	6.89	
3 - 2	-0.662	17.43				
4 - 1	-0.662	25.33	25.33	---	7.01	
4 - 2	-0.662	23.50				

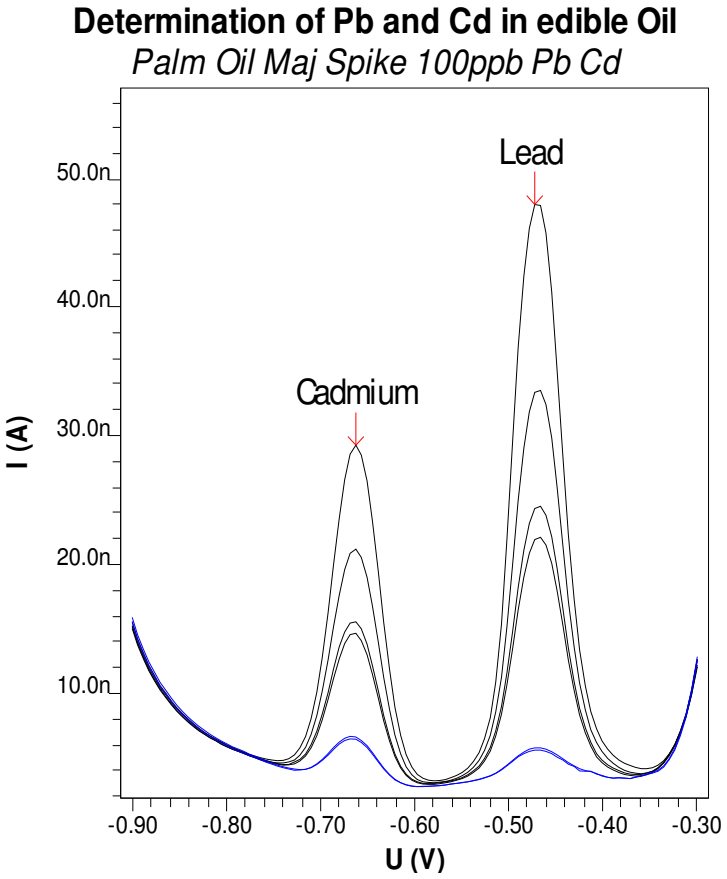
Substance : Lead
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.471	2.61	2.61	---	0.00	
1 - 2	-0.471	2.45				
2 - 1	-0.466	21.01	19.84	1.650	17.23	
2 - 2	-0.466	18.68				
3 - 1	-0.466	34.63	32.25	3.365	12.40	
3 - 2	-0.466	29.87				
4 - 1	-0.471	43.99	43.99	---	11.74	
4 - 2	-0.466	38.62				

Solutions

No.	Content	Predose (mL)
1	Standard For Cd and Pb	-------

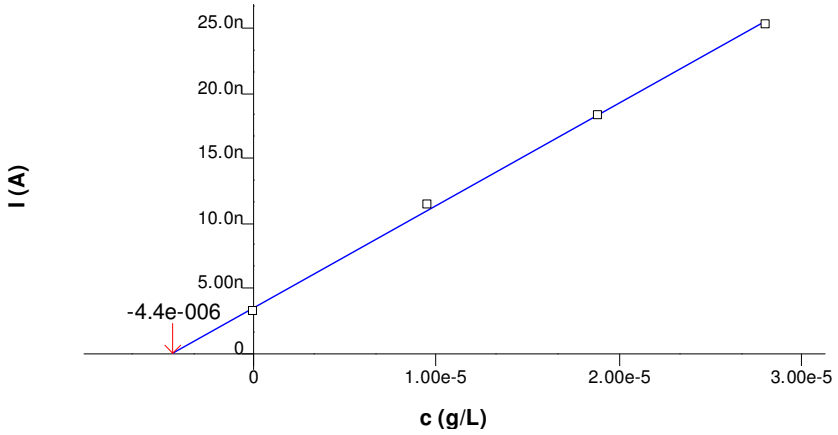
Final results			+/- Res. dev.	%	Comments
Cadmium:					
default	=	91.567 ug/L	12.549	13.705	
Lead :					
default	=	43.927 ug/L	14.266	32.476	



Cadmium

c = 91.567 ug/L

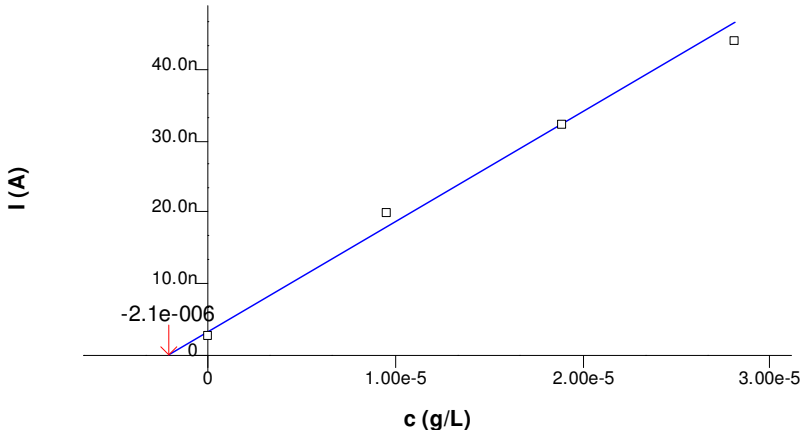
+/- 12.549 ug/L (13.71%)



Lead

c = 43.927 ug/L

+/- 14.266 ug/L (32.48%)



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Oil Maj Spike 100ppb Ni.dth
Sample ID : Palm Oil Maj Spike 100ppb Ni
Creator determ.: Date : 2009-12-29 Time: 11:03:42
Modified by : Date : 2010-01-20 Time: 17:39:41

Method : Ni Determination in Edible Oil (continous method).mth
Title : Ni Determination in Edible Oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : Depostion time 5s, total volume 14.75

Sample amount : 0.500 mL
Cell volume : 14.750 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.966	-48.9	-49.4	---	0.0	Not used
1 - 2	-0.966	-49.4				
2 - 1	-0.970	-86.8	-87.1	0.439	-37.7	
2 - 2	-0.970	-87.4				
3 - 1	-0.970	-121.7	-123.6	2.784	-36.6	
3 - 2	-0.970	-125.6				
4 - 1	-0.970	-158.9	-158.1	1.125	-34.4	
4 - 2	-0.970	-157.3				

Solutions

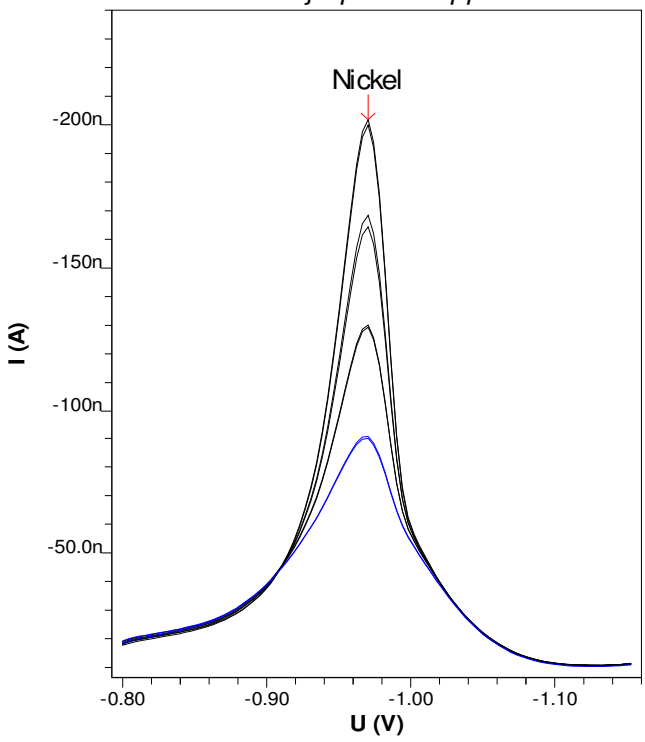
No.	Content	Predose (mL)

Final results	+/-	Res. dev.	%	Comments

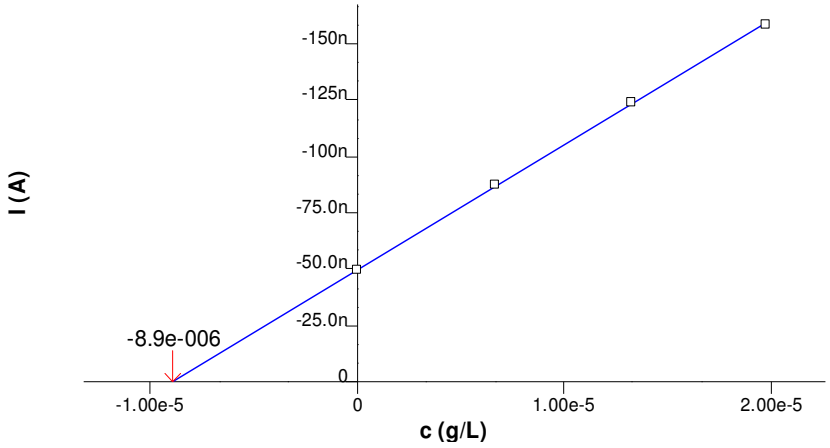
Nickel:
default = 263.234 ug/L 4.381 1.664

Ni Determination in Edible Oil

Palm Oil Maj Spike 100ppb Ni



Nickel
c = 263.234 ug/L
+/- 4.381 ug/L (1.66%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Palm Oil Maj Spike 100ppb Zn .dth
Sample ID : Palm Oil Maj Spike 100ppb Zn
Creator determ .: Date : 2009-12-29 Time: 10.45.02
Modified by : Date : 2010-01-20 Time: 18.00.13

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl continous from previous Cd pb)
Remark2 : Deposition time 90s, total volume 10.65.

Sample amount : 0.500 mL
Cell volume : 10.650 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-1.100	12.33	12.43	0.141	0.00	
1 - 2	-1.100	12.53				
2 - 1	-1.100	22.49	22.49	0.013	10.05	
2 - 2	-1.100	22.48				
3 - 1	-1.094	30.69	30.38	0.440	7.90	
3 - 2	-1.094	30.07				
4 - 1	-1.094	37.70	37.82	0.167	7.44	
4 - 2	-1.094	37.94				

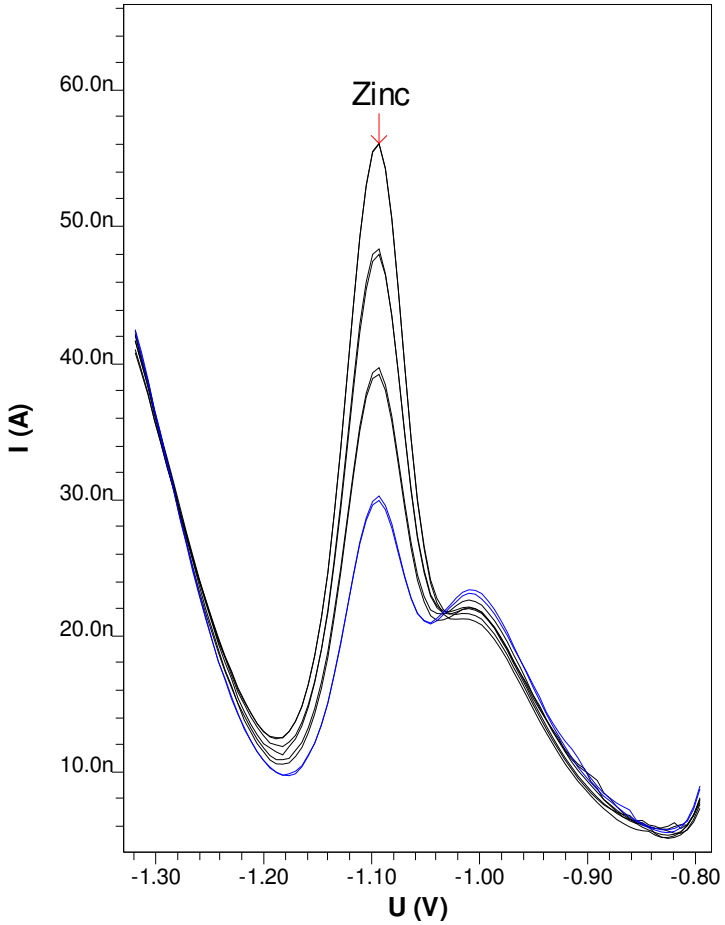
Solutions

No.	Content	Predose (mL)

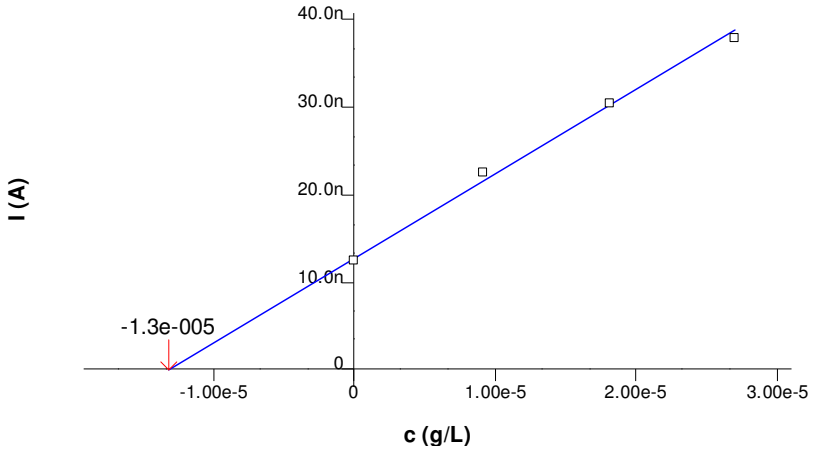
Final results	+/-	Res. dev.	%	Comments
Zinc:				
default = 280.463 ug/L		14.152	5.046	

Determination of Zn in edible Oil

Palm Oil Maj Spike 100ppb Zn



Zinc
c = 280.463 ug/L
+/- 14.152 ug/L (5.05%)



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Sunflower Maj Pb,Cd.dth
Sample ID : Sunflower Maj Pb Cd
Creator determ.: Date : 2009-12-30 Time: 13:21:29
Modified by : Date : 2010-01-21 Time: 21:04:06

Method : Determination of Cd and Pb in edible Oil with Acetate buffer
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.50 smpl + 10ml UPW)+0.25ml Acetate buffer
Remark2 : Deposition time 30s,

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.704	1.15	1.00	0.218	0.00	
1 - 2	-0.698	0.84				
2 - 1	-0.698	12.23	10.49	---	9.49	
2 - 2	-0.698	10.49				
3 - 1	-0.698	17.71	17.42	0.416	6.93	
3 - 2	-0.698	17.13				
4 - 1	-0.698	24.24	23.74	0.698	6.33	
4 - 2	-0.698	23.25				

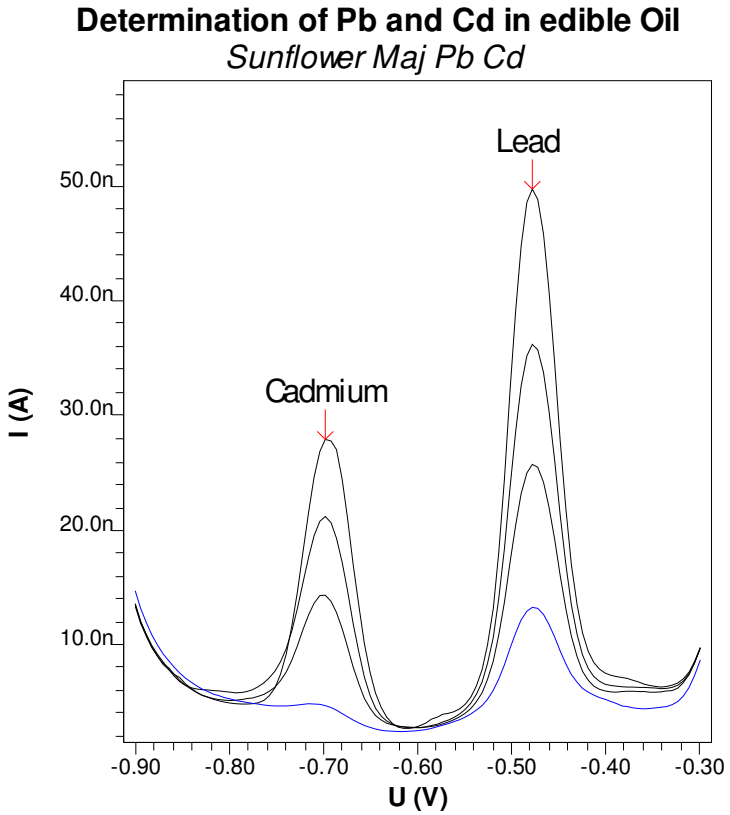
Substance : Lead
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.477	9.82	9.13	0.986	0.00	
1 - 2	-0.477	8.43				
2 - 1	-0.477	23.79	21.60	---	12.48	
2 - 2	-0.477	21.60				
3 - 1	-0.477	35.96	33.95	2.840	12.35	
3 - 2	-0.477	31.94				
4 - 1	-0.477	45.39	42.09	4.670	8.14	
4 - 2	-0.477	38.79				

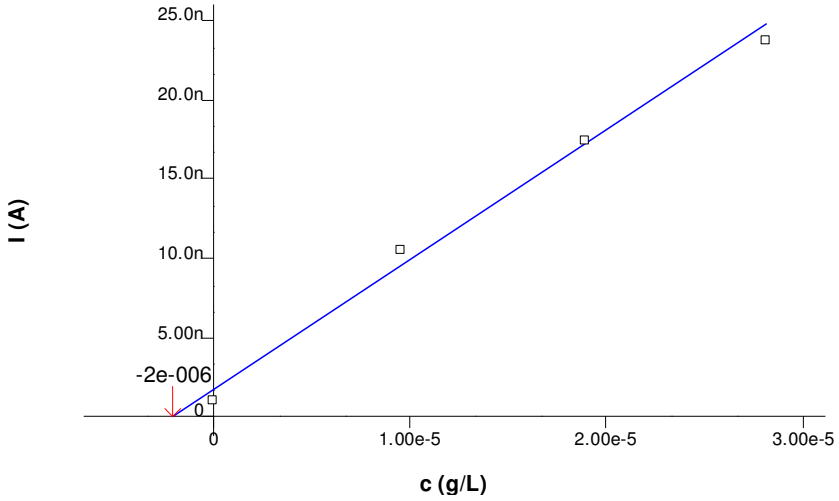
Solutions

No.	Content	Predose (mL)
1	Standard For Cd and Pb	-------

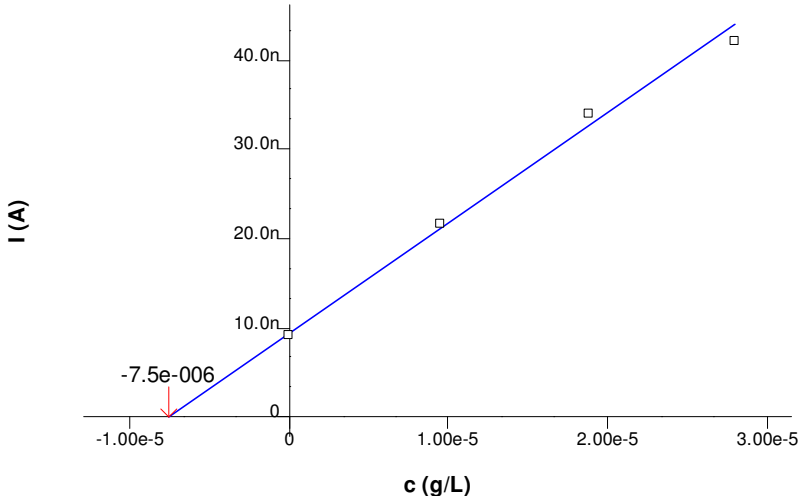
Final results	+/-	Res. dev.	%	Comments
Cadmium:				
default = 42.374 ug/L		15.347	36.217	
Lead :				
default = 156.246 ug/L		19.955	12.771	



Cadmium
c = 42.374 ug/L
+/- 15.347 ug/L (36.22%)



Lead
c = 156.246 ug/L
+/- 19.955 ug/L (12.77%)



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Sunflower Maj Ni.dth
Sample ID : Sunflower Maj Ni
Creator determ.: Date : 2009-12-30 Time: 14:07:03
Modified by : Date : 2010-01-21 Time: 21:46:13

Method : Ni Determination in Edible Oil (continous method).mth
Title : Ni Determination in Edible Oil
Remark1 : Sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : deposition time 5s, total volume 14.75

Sample amount : 0.500 mL
Cell volume : 14.750 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.970	-57.4	-57.7	0.450	0.0	
1 - 2	-0.970	-58.0				
2 - 1	-0.970	-97.7	-96.6	1.643	-38.8	
2 - 2	-0.970	-95.4				
3 - 1	-0.970	-135.9	-136.1	0.328	-39.6	
3 - 2	-0.970	-136.3				
4 - 1	-0.970	-168.3	-166.4	2.681	-30.3	
4 - 2	-0.970	-164.5				

Solutions

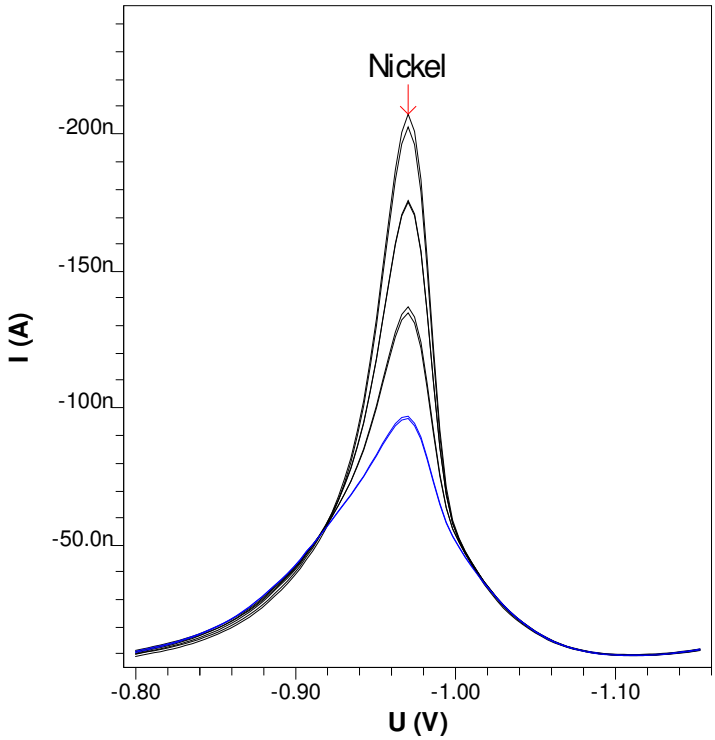
No.	Content	Predose (mL)

Final results	+/-	Res. dev.	%	Comments

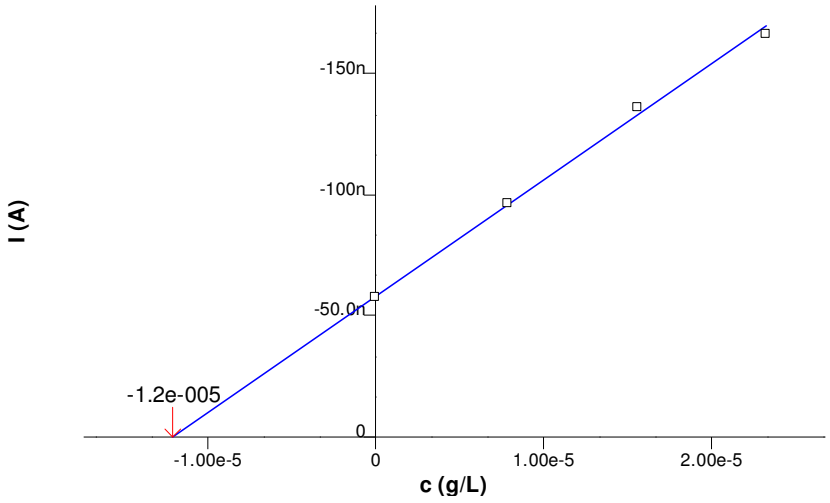
Nickel:
default = 300.222 ug/L 9.340 3.111

Ni Determination in Edible Oil

Sunflower Maj Ni



Nickel
c = 300.222 ug/L
+/- 9.340 ug/L (3.11%)



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Sunflower Maj Zn .dth
Sample ID : Sunflower Maj Zn
Creator determ.: Date : 2009-12-30 Time: 13:46:41
Modified by : Date : 2010-01-21 Time: 21:15:18

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl continous from previous Cd pb)
Remark2 : Deposition time 90s, total volume 10.65

Sample amount : 0.500 mL
Cell volume : 10.650 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 250.00 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-1.010	8.52	8.85	---	0.00	
1 - 2	-1.010	8.85				
2 - 1	-1.004	32.99	32.04	1.344	23.19	
2 - 2	-1.004	31.09				
3 - 1	-1.010	54.14	53.11	1.454	21.07	
3 - 2	-1.004	52.08				
4 - 1	-1.004	72.18	72.10	0.111	18.99	
4 - 2	-1.004	72.02				

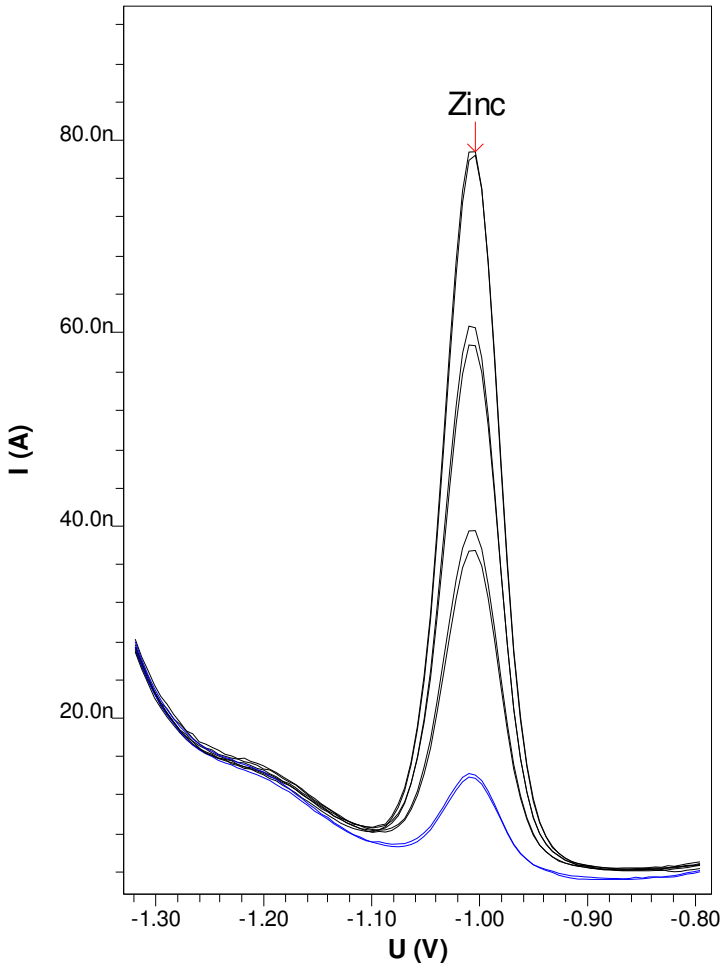
Solutions

No.	Content	Predose (mL)

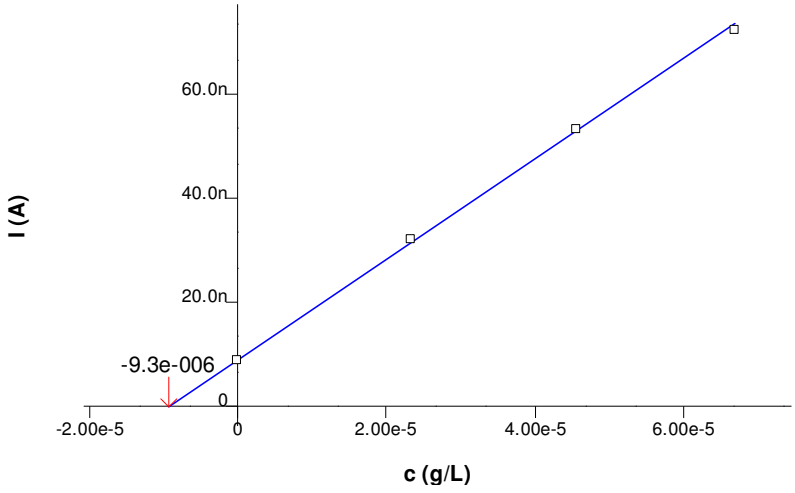
Final results	+/-	Res. dev.	%	Comments
Zinc:				
default = 192.074 ug/L		8.010	4.170	

Determination of Zn in edible Oil

Sunflower Maj Zn



Zinc
c = 192.074 ug/L
+/- 8.010 ug/L (4.17%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158) ==========

Determination :Sunflower Oil Cd Pb.dth
Sample ID : Sunflower Oil Cd Pb
Creator determ.: Date : 2009-12-24 Time: 11:20:36
Modified by : Date : 2010-01-22 Time: 14:57:09

Method : Determination of Cd and Pb in edible Oil with Acetate buffer
Title : Determination of Pb and Cd in edible Oil
Remark1 : Sample (0.50 smpl + 10ml UPW)+0.25ml Acetate buffer
Remark2 : Deposition time 30s,

Sample amount : 0.500 mL
Cell volume : 10.350 mL

Substance : Cadmium
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.702	9.67	9.48	0.293	0.00	
1 - 2	-0.684	9.14				
1 - 3	-0.702	9.63				
2 - 1	-0.661	21.16	21.82	1.368	12.34	
2 - 2	-0.661	20.86				
2 - 3	-0.667	22.79				
3 - 1	-0.661	29.18	30.56	1.940	8.73	
3 - 2	-0.667	31.93				
3 - 3	-0.672	34.09				

Substance : Lead
Conc. : 1.000 mg/L
Conc.dev. : ---
Add.amount : 100.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-0.458	2.77	2.67	0.133	0.00	
1 - 2	-0.458	2.58				
1 - 3	---	---				
2 - 1	-0.452	8.28	8.70	0.406	6.03	
2 - 2	-0.452	8.42				
2 - 3	-0.452	8.99				
3 - 1	-0.446	15.23	16.69	2.068	7.99	

3 - 2 -0.446 18.15
3 - 3 -0.452 19.42

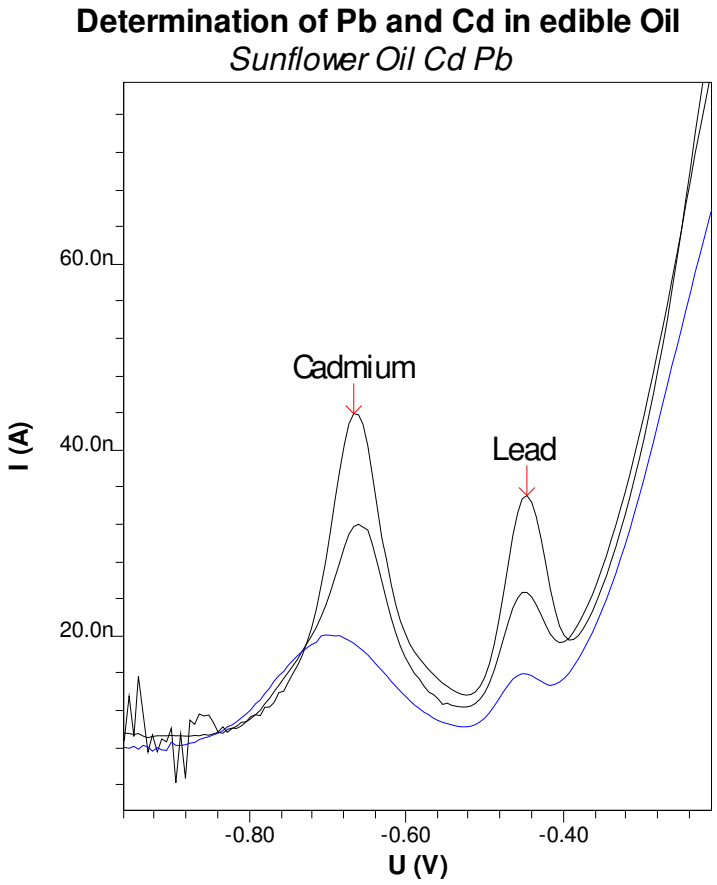
Solutions

No.	Content	Predose (mL)
---	---	---

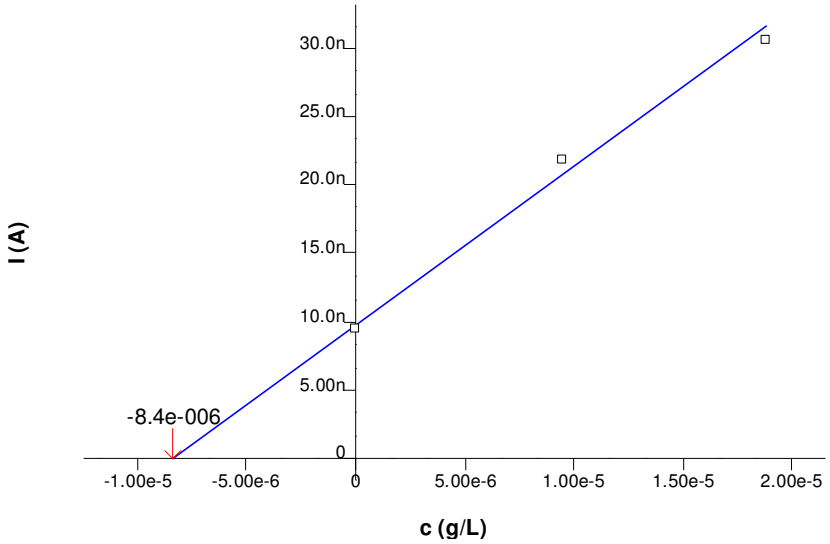
Final results	+/-	Res. dev.	%	Comments
---	---	---	---	---

Cadmium:
default = 173.065 ug/L 14.831 8.569

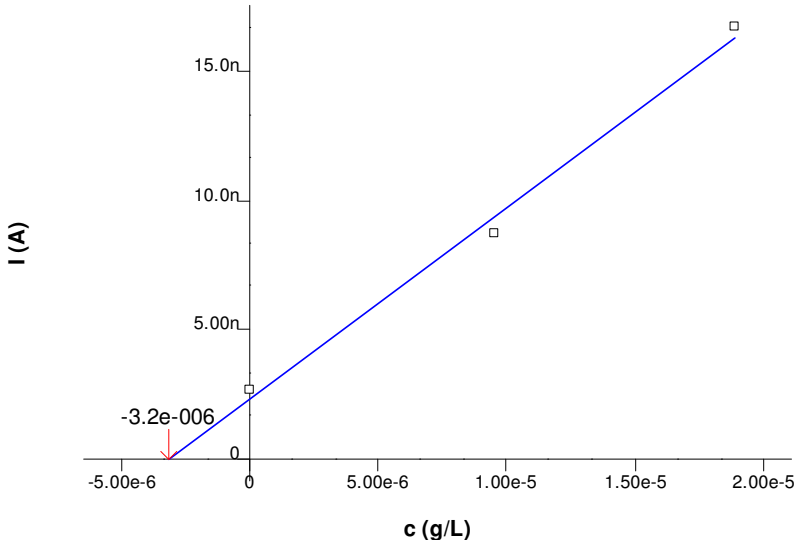
Lead :
default = 65.663 ug/L 17.678 26.923



Cadmium
c = 173.065 ug/L
+/- 14.831 ug/L (8.57%)



Lead
c = 65.663 ug/L
+/- 17.678 ug/L (26.92%)



========== METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Sunflower Oil Ni.dth
Sample ID : Sunflower Oil Ni
Creator determ.: Date : 2009-12-24 Time: 12.02.03
Modified by : Date : 2010-01-22 Time: 15:31:30

Method : Ni Determination in Edible Oil (continous method).mth
Title : Ni Determination in Edible Oil
Remark1 : sample 0.5ml Samp+ 1.0ml NH4Cl Buffer + 0.1ml DMG
Remark2 : deposition time 5s, Total Volume 11.400ml

Sample amount : 0.500 mL
Cell volume : 11.400 mL

Substance : Nickel
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 30.000 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-1.022	-9.82	-9.95	0.428	0.00	
1 - 2	-1.022	-9.61				
1 - 3	-1.022	-10.43				
2 - 1	-1.038	-18.85	-19.56	1.675	-9.61	
2 - 2	-1.038	-21.48				
2 - 3	-1.038	-18.36				
3 - 1	-1.038	-28.96	-29.27	0.740	-9.71	
3 - 2	-1.038	-30.12				
3 - 3	-1.038	-28.74				

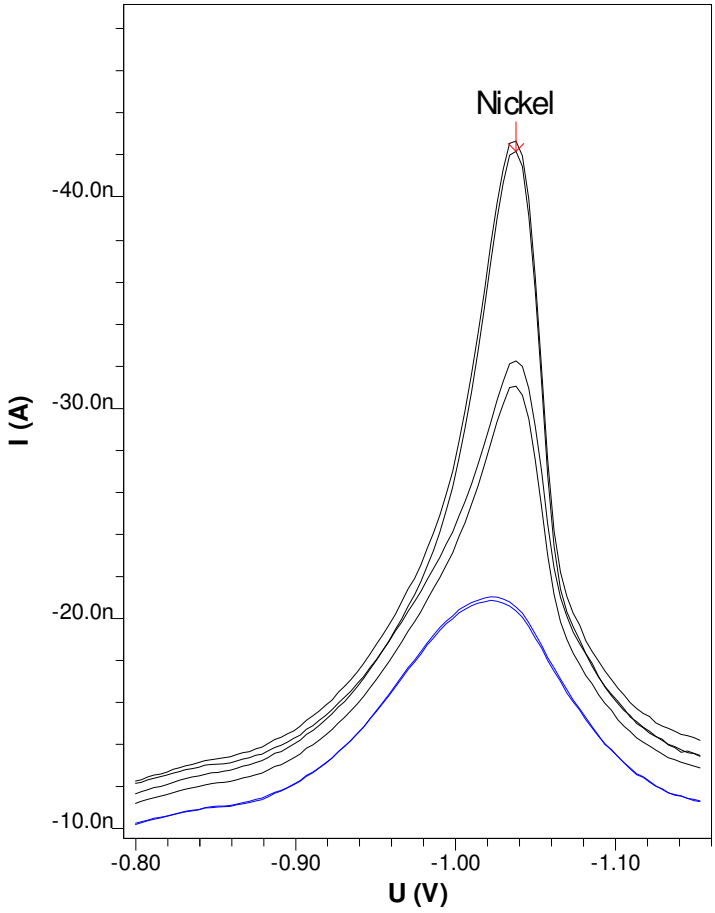
Solutions

No.	Content	Predose (mL)

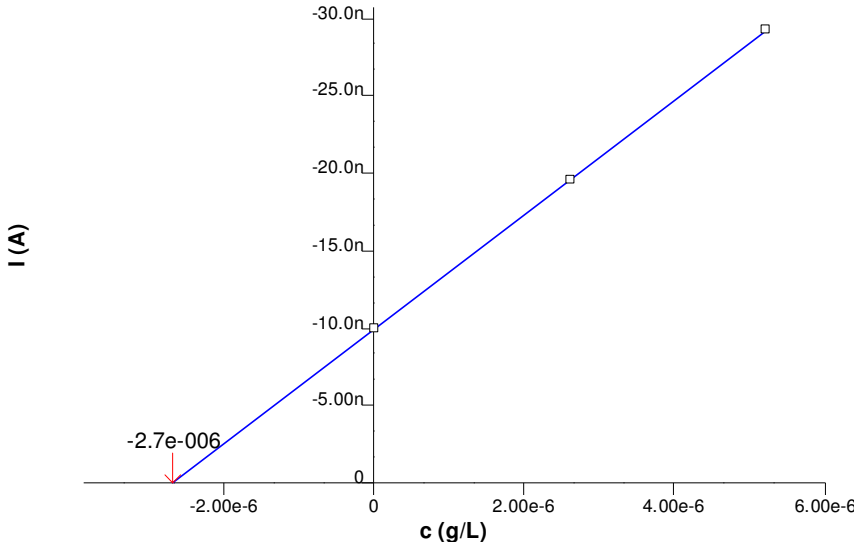
Final results	+/-	Res. dev.	%	Comments
Nickel:				
default = 61.381 ug/L	3.695		6.020	

Ni Determination in Edible Oil

Sunflower Oil Ni



Nickel
c = 61.381 ug/L
+/- 3.695 ug/L (6.02%)



========= METROHM 797 VA COMPUTRACE (Version 1.2) (Serial No. 3158)
==========

Determination : Sunflower Oil Zn .dth
Sample ID : Sunflower Oil Zn
Creator determ.: Date : 2010-01-18 Time: 11:49:23
Modified by : Date : 2010-01-22 Time: 15:19:52

Method : Determination of Zn in edible Oil (Continous).mth
Title : Determination of Zn in edible Oil
Remark1 : Sample (0.50 smpl + 10ml UPW)+0.25ml Acetate buffer
Remark2 : Deposition time 90s, total volume 10.35 - repeat

Sample amount : 0.500 mL
Cell volume : 10.35 mL

Substance : Zinc
Conc. : 1.000 mg/L
Conc.dev. :
Add.amount : 250.00 ul

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	-1.004	18.49	19.43	1.332	0.00	
1 - 2	-1.004	20.37				
2 - 1	-1.004	39.45	41.23	2.510	21.79	
2 - 2	-1.004	43.00				
3 - 1	---	---	63.89	---	22.66	
3 - 2	-1.004	63.89				
4 - 1	-1.004	77.60	79.61	2.850	15.72	
4 - 2	-1.004	81.63				

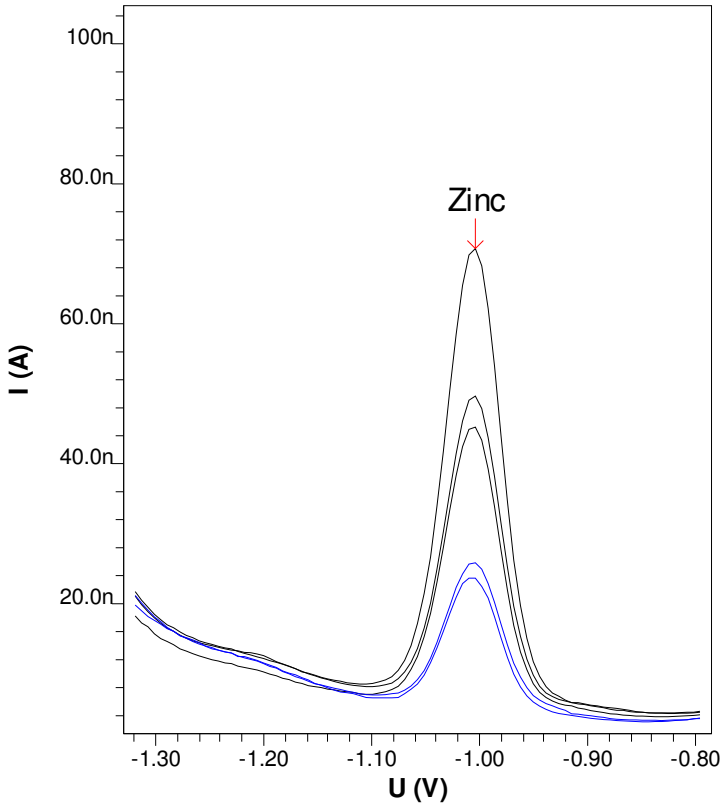
Solutions

No.	Content	Predose (mL)

Final results	+/-	Res. dev.	%	Comments
Zinc:				
default = 429.058 ug/L		25.247	5.884	

Determination of Zn in edible Oil

Sunflower Oil Zn



Zinc
c = 429.058 ug/L
+/- 25.247 ug/L (5.88%)